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**WORK PLAN** 

FOR A

FIELD INVESTIGATION

AT

THE SELMER COMPANY 500 INDUSTRIAL PARKWAY ELKHART, INDIANA

Prepared for

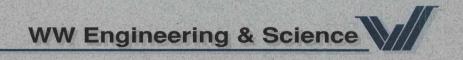
NORTH AMERICAN PHILIPS CORPORATION
THE SELMER COMPANY
AND
MACMILLIAN, INC.

Prepared by

WW ENGINEERING & SCIENCE 5555 GLENWOOD HILLS PARKWAY GRAND RAPIDS, MICHIGAN 49588-0874

**AUGUST 1992** 

PROJECT 22334



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#### 1.0 INTRODUCTION

WW Engineering & Science (WWES) has been jointly retained by North American Philips Corporation, The Selmer Company and Macmillian, Inc. to prepare and implement an investigation work plan for The Selmer Company facility located at 500 Industrial Parkway in the Eastside Industrial Park in Elkhart, Indiana (see Figure 1). The Selmer Company facility, hereinafter referred to as the "facility", was built in 1965 and has been used exclusively for the manufacturing of brass musical instruments. Solvents are used during the manufacturing process to degrease and clean metal parts between plating operations. The purpose of the investigation is a) to determine the identity, amounts and location of hazardous substances, pollutants or contaminants in the environment at the facility, and b) to evaluate alternatives for any appropriate remedial action to prevent, mitigate or otherwise remedy any release or threatened release of any hazardous substances, pollutants or contaminants into the environment at the facility. Governmental and private-party technical investigations have reported detectable VOCs in the ground water in eastern Elkhart since 1976.

#### 1.1 GENERAL GEOLOGY AND HYDROGEOLOGY

The plant is located within the St. Joseph River basin between the St. Joseph River and the Elkhart River, a tributary of the St. Joseph River (see Figure 1). The St. Joseph River and the Elkhart River flows to the west and northwest, respectively. The physiography of the region ranges from flat plains to rolling and hilly terrain. The study property is relatively flat with the exception of two topographic depressions which are located east and south of the facility.

#### 1.1.1 GEOLOGY

Geology of the area consists of approximately 120 feet of sand and gravel outwash deposits interbedded with a confining silt and clay unit overlying bedrock (Imbrigiotta and Martin, 1981). The thick deposit of stratified and unstratified drift was deposited in the Pleistocene Epoch during four glacial advances and retreats (Imbrigiotta and Martin, 1981). According to Johnson and Keller (1972) and Schneider and Keller (1970), the bedrock consists of the Coldwater Shale of Mississippian age and the Sunbury and the Ellsworth Shales of Devonian and Mississippian age.

Based on soil borings drilled to a maximum depth of 59 feet, the plant property geology appears to consist of 26 to 52 feet of medium to course sand or sand and gravel overlying gray silty clay. A dark brown loamy sand was observed 1 to 3 feet below ground surface for the majority of the property except in the vicinity of the small shed located east of the manufacturing building. In this area, a sand fill of varying thickness of 3.5 to 12 feet was observed.

#### 1.1.2 HYDROGEOLOGY

According to Imbrigiotta and Martin (1981), the confining layer of silt and clay within the sand and gravel outwash deposit is 60 feet thick in the western half of the industrial park. The outwash deposit overlying the silt and clay confining layer is of average thickness 40 feet, is areally extensive, and locally exists as an unconfined aquifer. The outwash deposit between the confining silt and clay layer and bedrock is approximately 20 feet thick and exists under confined conditions.

The regional ground water flow is generally horizontal and towards the river systems. Vertical components of flow are minimal, except in areas near the major streams, where upward gradients exist. This flow pattern is characteristic of a well-connected stream-aquifer system with a gaining stream. Ground water flow at the facility most likely flows to the west or northwest. Ground water was encountered at depths ranging from 1 foot to 14 feet during drilling of soil borings at the plant in 1989.

The U.S.G.S. study of the region as described in Section 1.4.2 noted an annual fluctuation of 2 to 4 feet in the water table (Imbrigiotta and Martin, 1981). The fluctuation was attributed to variation in recharge to the aquifer.

#### 1.2 DESCRIPTION OF INDUSTRIAL PARK

Eastern Elkhart includes a 500-acre industrial park, the Eastside Industrial Park, which is bounded on the north by Conrail tracks, on the west by Riverview Avenue and Outer Drive, on the south by U.S. 20, and on the east by Middletown Run Road (see Figure 2). The Selmer facility is located in the northwest quadrant of the industrial park.

#### 1.3 DESCRIPTION OF FACILITY

The facility consists of 18.45 acres of lightly wooded land and is located in the W1/2 of SE1/4 of Section 3 of T37N, R5E of the Elkhart Quadrangle. The main manufacturing

building and associated storage buildings are located in the northern half of the property (see Figure 3). An office building with a parking lot is located at the south end of the facility. Asphalt pavement for parking also exists along the western portion of the facility.

Surface water runoff drains to topographic depressions located east of the manufacturing building and west of the office building, which are wooded and periodically contain standing water.

The facility, currently operating under the name "Vincent Bach Company", was constructed in 1965. The facility was operated from 1965 to 1970 by C.G. Conn, Ltd. The property was transferred in June 1990 to The Selmer Company. On December 29, 1988, The Selmer Company was sold to Integrated Resources Inc., the current owner.

Several additions have been added to the manufacturing facility since its construction in 1965. In 1971, the building was expanded approximately 59,000 square feet and 12,000 square feet to the north and east of the original building, respectively. In 1972, a southern extension of approximately 15,000 square feet was added to the original facility.

The facility has been used exclusively for the manufacture of brass musical instruments. Solvents are used during the manufacture of band instruments to degrease and clean brass components prior to finishing with lacquer and to clean the parts in assembly operations. Trichloroethylene (TCE) is a solvent used during the manufacturing process.

Since the facility began operating in 1965, degreasing operations have occurred chiefly in vapor phase degreasers (VPDs). VPDs were connected to solvent stills to reclaim the used solvent for reuse. The solvent stills on the VPDs generate sludge, commonly referred to as "still bottoms", which must be removed periodically from the stills. The still bottoms consist chiefly of animal fats, buffing compounds, and a small percentage by weight of solvents (TCE). The still bottoms generated from the manufacturing process were stored in 55-gallon drums for transport off-site to a solvent recovery firm.

#### 1.4 PREVIOUS STUDIES OF EASTERN ELKHART AREA

Laboratory analyses of ground water samples obtained throughout the eastern Elkhart area have documented the occurrence of several VOCs, including TCE, TCA, and

dichloromethane (methylene chloride). TCE, which is commonly used as a degreaser by many industries, is the principal VOC detected in the area.

VOCS were first detected in ground water by the U.S. EPA in 1976. Four shallow residential wells located between the St. Joseph River and the industrial park were sampled following reports from residents of taste and odor problems in the drinking water. Eight VOCS were detected in the residential wells including methylene chloride, 1,1-dichloroethane (DCA), and TCA. The lateral extent of impact on the local ground water system was not determined.

In 1979, the U.S. Geological Survey (USGS) conducted a hydrologic and chemical evaluation of the ground water resources of northwest Elkhart County (Imbrigiotta and Martin, 1981). One of the objectives of the study was to characterize the ground water flow regime and the ground water quality of the area. The scope of work included organic laboratory analyses of ground water samples collected from 19 monitoring wells located within the industrial park and the residential area north of the park. The 19 monitoring wells were screened at depths of 45 feet or less. One of the 19 wells sampled was located at the northwest corner of the Selmer Company facility. No VOCS were detected in the ground water samples collected from this well.

VOCS were detected in ground water samples collected from 8 of the 19 wells monitoring wells. Of these eight wells five were located within the residential area east of Superior Road, and three wells were located within the northeast corner of the industrial park.

The VOCs detected included TCE, TCA, methylene chloride, trichlorofluoromethane, trichloromethane (chloroform), DCA, methylbenzene (toluene), and 1,2-dichloropropane. However, only three compounds (TCE, TCA, and methylene chloride) were detected within the Eastside Industrial Park. Notably, the highest average concentration of all organic compounds were observed in wells located in the residential area north of the Conrail tracks and northeast of the facility. A source(s) of the VOCs could not be identified and a plume(s) could not be delineated based on the results of the USGS study.

In 1985 Weston Consultants, Inc. (WESTON-SPER, 1986), under contract to the U.S. EPA, conducted extensive ground water sampling of the private residential and industrial wells in the eastern Elkhart area. Within the industrial park and the residential area to the north, 142 water wells were analyzed for VOCs. TCE was the predominant compound

detected in the wells. The Weston study confirmed the presence of VOCs in the ground water in eastern Elkhart area, however, no sources for the VOCs were identified.

#### 2.0 PURPOSE AND SCOPE OF WORK

The primary purpose of the proposed investigation is to characterize the soil conditions at the facility to determine the presence or absence of an area(s) of contamination that may have resulted from the albert disposal of TCE. The objectives of the investigation are as follows:

- to better define the soil stratigraphy of the facility;
- to evaluate the occurrence and relative magnitude of VOCs that may be present in soil vapor in the unsaturated zone;
- if elevated VOCs are detected in the soil vapor, to quantify the VOCs in the soil by laboratory analyses;
- to delineate the impact of VOCs, if any, to soil at the facility; and
- to confirm the presence or absence of possible source areas on the plant property.

In order to meet these objectives, the investigation is proposed to be conducted in two phases. The initial phase will involve the implementation of a soil gas survey to identify potential source areas on the plant property. The scope of work for the Phase I will include PETREX<sup>©</sup> passive sampling techniques offered by the Northeast Research Institute (NERI) of Farmington, Connecticut.

The second phase will involve the implementation of a drilling and soil sampling program, if elevated VOCs are detected during the Phase I work. The Phase II work scope will include the (1) drilling of soil borings, (2) collection of soil samples during drilling for chemical testing, (3) laboratory analysis of selected soil samples for VOCs, and (4) interpretation of the analytical results. Based on the outcome of the Phase II work, it may become necessary to evaluate alternatives for any appropriate remedial action.

#### 3.0 PHASE I - SOIL GAS SURVEY

An assessment of the chemical characteristics of the soil vapor at the facility is proposed for the first phase of the investigation. The required scope of work for a second phase of investigation will be determined based on the results of the Phase I investigation.

The purpose of the Phase I work is to determine the presence or absence of VOCs in the subsurface which may have resulted from the alleged improper disposal of solvents historically. The scope of work will include the implementation of a soil gas survey at the facility. The use of soil gas technology provides the most effective means for identification of potential source areas at the facility.

#### 3.1 INTRODUCTION

The following two sections describe the PETREX<sup>©</sup> technology and the personnel that will be used to implement the PETREX techniques for the proposed Phase I work.

#### 3.1.1 Petrex Technology

A soil gas survey will be conducted using the high resolution soil gas technique known as the PETREX<sup>©</sup> Technology. The PETREX<sup>©</sup> method takes advantage of the recent advances in sorbent technology, collection device design, mass spectrometry, and computerized pattern recognition techniques (Einhorn, I. N. and others, 1991). The collection device design uses passive sampling techniques in which samples are collected from undisturbed soils. Passive soil gas sampling allows an equilibrium to develop between the soil gases and the sorbent, a charcoal device.

The charcoal adsorbent is adhered to two ferromagnetic wires within a glass tube. One wire is used for mass spectrometer (MS) analysis, the other wire is reserved for gas chromatograph/mass spectrometer (GC/MS) analysis, if needed. The passive charcoal devices are buried in shallow soil for a number of days and retrieved for analysis by desorption into the ion source of a MS via Curie-point thermal desorption. Refer to Appendix A for a more detailed account of the protocol followed for PETREX<sup>©</sup> soil gas surveys.

#### 3.1.2 PERSONNEL

The field and office work conducted by WWES personnel will be initiated from the Grand Rapids, Michigan office. WWES' Project Manager, Scott Dennis, and Project

Hydrogeologist, Lauryl Lefebvre, will assist in the coordination and implementation of the field and report writing activities associated with the project.

All field work will be conducted by properly trained personnel in accordance with Occupational Safety and Health Administration (OSHA) guidance and will be implemented in accordance with the Health & Safety Plan presented in Appendix B.

Prior to the installation of the gas samplers, the soil gas sample locations will be established by WWES' certified survey personnel. The PETREX<sup>©</sup> passive collectors will be installed, activated and removed at the facility by WWES field personnel trained in PETREX<sup>©</sup> soil gas procedures. Duplicate samplers and trip blanks will also be collected by WWES field personnel as part of the quality assurance/quality control (QA/QC) plan for the project.

Analytical testing of the collectors will be conducted by NERI's Farmington, Connecticut or Lakewood, Colorado laboratory. A summary and assessment of the analytical results will be provided by NERI's Connecticut office. This information will be included in WWES' Investigation Report.

#### 3.2 FIELD METHODS

This section summarizes the field methodology to be implemented during the Phase I investigation. For a more detailed description of field and laboratory protocol, refer to the PETREX<sup>©</sup> soil gas survey standard operating procedures (SOP's) presented in Appendix A.

#### **3.2.1 SURVEY**

The surveyors will establish a grid based on arbitrary local grid coordinates (assumed). The grid will include established and monumented control lines within an approximate 400- by 250-foot grid area (see Figure 4). A wood stake will be set at 50-foot grid intervals to locate all soil gas sampler locations.

The surveyors will locate all soil gas points relative to the grid. All locations of the soil gas samplers will be recorded with a 1.0-foot accuracy. A map has been created based on a composite map (aerial photo of Spring 1986 plus plat map) of the S1/2, SE1/4, Section 3, T37N, R5E of Concord Township (see Figure 3). The sampler locations will be plotted with respect to this map.

In addition, several benchmarks will be established by the WWES' surveyors at the third order accuracy based on the National Geodetic Vertical Datum (NGVD) of 1929. The benchmarks will be used to establish ground surface elevations for all soil gas sampler locations. All elevations will be recorded to the nearest one-tenth of a foot.

#### 3.2.2 Installation of Samplers

A total of 67 PETREX<sup>©</sup> soil gas samplers, provided by NERI, will be placed in uniform arrays (see Figures 3 and 4). A grid with a spacing of 50 feet will surveyed within an approximate 400-foot by 250-foot area east of the manufacturing facility. Within the 50-foot grid, a more closely spaced 25-foot grid pattern will be surveyed in the area immediately east of the original manufacturing plant. The sample locations will be labeled with the prefix "SG" as indicated in Figure 4. Sample locations may be slightly altered depending on accessibility.

The passive collectors will be activated in the field by removing the cap and seal and placing them in an inverted position into cored holes at a depth of 17 inches. The boreholes will be drilled with a hammer drill using a 1.5-inch by 18-inch bit to accommodate the 1-inch outer diameter (OD) sampler. The boreholes will be backfilled with native soil cuttings, and flagged for easy location. At each sample location, field notes will be recorded regarding sample location, type of sampler installed (regular or duplicate), date and time of installation, soil profile, type of backfill, moisture conditions, type of flagging, and staining of soil.

For the samplers located within areas of asphalt pavement or concrete, the boreholes may be backfilled with crushed aluminum foil to one inch below grade. The remaining one inch will be backfilled with quick-setting cement. For the samplers located within the periodically swampy area east of the manufacturing facility, the samplers will be encapsulated in a Ziplock bag prior to installation; the Ziplock bag is impermeable to water, yet permeable to VOCs. Additional attention will be given to flagging the stations within the swampy areas to ensure sample retrieval.

#### 3.2.3 REMOVAL OF SAMPLERS

All soil gas samplers will be installed within a 24 to 72 hour time period and will be retrieved after a maximum 28-day residence period. During the residence period, the samplers will equilibrate with the soil vapors of the undisturbed medium. Upon retrieval, the gas samplers will be shipped to NERI's Lakewood, Colorado laboratory for MS

analysis. If GC/MS analysis is required for separating coelution species of a complex mixture of pollutants, the sampler will be forwarded to NERI's Farmington, Connecticut laboratory.

At the time of installation, two time calibration samplers in addition to a regular sampler will be installed at two sample locations at the facility. One set of time calibration samplers, TC-1, will be installed near the shed east of the plant. A second set of the time calibration samplers, TC-2, will be installed at a location just north of the low wooded area. One time calibration sampler from each set will be retrieved three days from the time of installation. The remaining time calibration sampler at sample locations TC-1 and TC-2 will be retrieved seven days from the time of installation.

The purpose of the time calibration samplers is to assess the loading rate of VOCs onto the Petrex<sup>©</sup> collector wires. The results obtained from the time calibration samplers may reduce the total residence time for the survey gas samplers.

#### 3.3 LABORATORY TESTING

Upon retrieval of the soil vapor survey samplers from the field, the samplers will be returned to the laboratory for a full VOC scan. The analyses will be performed by Curie-point desorption directly into the ion source of an interfaced quadrupole MS. During each sample analysis, the desorbed VOCs will normally be analyzed in the mass range of 30 to 240 amu. The information obtained during the analysis process will be stored in the computer as a composite of the VOCs collected at each sample location. The data will be downloaded onto a graphics workstation where the information will be processed using a variety of chemometric techniques.

Identification and relative response of the volatile organic compounds present with the gas sampler will be reported by the analytical laboratory. Compound identification will be based on molecular weight, compound fragmentation, and isotope distribution, as applicable. Relative response reported for each compound is based the observed ion count during sample analysis (Einhorn, N. and others, 1991).

#### 3.4 QA/QC PROGRAM

The QA/QC plan for the Phase I is based on WWES' standard field protocol and on the recommendations of NERI. The plan includes collecting duplicate samples and trip blanks during field activities and running method blanks during laboratory analyses. No

equipment blank samples will be collected since the soil gas samplers are dedicated to each sampling location. Additionally, reagent blanks will not be run during analytical testing since no reagents are required to perform the analysis.

#### 3.4.1 FIELD LOGBOOKS/DOCUMENTATION

Logbooks will used to record all data collection activities performed in the field. Entries in the field logbooks will be described in as much detail as possible so that persons returning to the facility may re-construct a particular situation without reliance upon memory. Each entry will be associated with a project-specific document number. All entries will be made in ink and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark, initialed and dated.

The title page of each logbook will contain the following:

- person to whom the logbook is assigned;
- logbook number;
- project name; and
- project start and end dates.

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all field team members present, level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors, field sampling, or investigation team personnel and the purpose of their visit will also be recorded in the field logbook. A guideline for taking field notes and records is included in Appendix C.

Although the field activities are proposed to be performed in accordance with the procedures documented in this work plan and in the PETREX<sup>©</sup> soil gas survey SOP. presented in Appendix A, the field logbook will note any necessary deviations. The field logbook will be used to record the date and time of installation and retrieval of each soil gas sampler, sampler number, sampler location description, soil type, and general observations. Soil gas samplers also serving as duplicate samplers will be noted in the logbook, in the sample identification, and in the chain-of-custody form.

#### 3.4.2 DUPLICATES

A total of six soil gas samplers installed during the survey will be duplicate samplers (one duplicate sample for every 10 samples collected). The duplicate samplers contain three

element wires, unlike the regular samplers which contain two element wires. The sample identification "SG-#" will be followed "(duplicate)" to indicate that a duplicate sampler was used at the sample location. Duplicate samplers will be installed at sample locations SG-10, SG-20, SG-30, SG-40, SG-50, and SG-60, as indicated on Figure 4.

The duplicate samplers will be collected for two reasons. First, the duplicate sample will provide the mass spectrometer operator with some measure as to the relative levels of compounds on the collectors. With this information, the operator may then set the instrument to optimum performance levels. This will be accomplished by analyzing the duplicate sample prior to the survey sample. Based on the results, the operator may slightly increase or decrease the sensitivity of the mass spectrometer. Secondly, the duplicate samples will show that the instrument is detecting the same compounds from the wires of both samples, although the level of intensity may vary.

The duplicate samples will be analyzed with identical machine parameters and compared with respect to compound identification of the original samples.

#### 3.4.3 TRAVEL BLANKS

Two PETREX<sup>©</sup> soil gas samplers will be included in this survey as travel blanks (two travel blanks for every shipment). The travel blanks will be labeled with the prefix "TB". The purpose of the travel blanks is to demonstrate that contamination was not introduce during transport of the survey samplers. The travel blank samplers will be sealed throughout the survey and will travel with the survey samplers.

The travel blanks will be analyzed with identical machine parameters and compared to the results of the survey samplers.

#### 3.4.4 SOIL GAS SAMPLER AND SHIPMENT

The sample packaging and shipment procedures summarized below will insure that the samples will arrive at the laboratory with the chain-of-custody intact. Examples of chain-of-custody forms and other field and sampling activity forms are located in Appendix D.

- WWES field personnel are responsible for the care and custody of the soil gas samplers until the samplers are transferred or properly dispatched. As few people as possible should handle the samplers.
- All soil gas samplers will be tagged with sample numbers and locations.

- Sample tags are to be completed for each sampler using waterproof ink unless prohibited by weather conditions.
- Once all soil gas samplers have been retrieved, they will be sealed in Ziplock bags, wrapped in bubble packing material, and packed tightly in a box for shipment.
- Samplers will be shipped by overnight courier service to NERI's analytical laboratory in Lakewood, Colorado. The shipment will be accompanied by a properly completed chain-of-custody record identifying the contents.
- If the soil gas samples are sent by common carrier, a bill of lading shall be used. Receipts of bills of lading will be retained as part of the permanent documentation. If sent by mail, the package will be registered with return receipt requested. Commercial carriers are not required to sign off on the custody form as long as the custody forms are sealed inside the sampler container.
- When transferring the possession of samplers, the individuals relinquishing and receiving the samplers will sign, date, and note the time on the record. This record documents transfer of custody of samplers from the field technician to another person, to the NERI laboratory, or to/from a secure storage area. The original record will accompany the shipment, and a copy will be retained by the field personnel and returned to the WWES project manager.

#### 3.4.5 LABORATORY TESTING

Quality assurance by NERI will be maintained by tuning the mass spectrometer with the internal standard perfluorotributylamine to obtain correct mass assignment and peak resolution. Periodic machine background analyses (approximately every 20 samples) will be performed to assure that there is no carry-over between successive samples. In addition, the mass spectrometer control program contains appropriate "flag statements" that prompt the operator with a warning if an input sample number has already been analyzed. The operator then checks the current number, along with the disk storage location of the previously entered number to identify the true numbering situation.

#### 3.5 REPORT

A report summarizing the results of soil gas survey will be prepared by WWES at the conclusion of all field and laboratory activities. Recommendations regarding the need for additional investigation will also be presented. If the implementation of a Phase II drilling and sampling program is warranted, the recommended location and depth of the soil borings will also be specified in the report.

#### 4.0 PHASE II - DRILLING AND SAMPLING PROGRAM

An additional examination of the soil conditions at the facility will be conducted if the Phase I results indicate the presence of elevated VOCs in the soil gas at the facility. The second phase would include the implementation of a drilling and sampling program. The purpose of the drilling and sampling program is to assess the impact of VOCs to the soil at the facility and to determine the source(s) of the impacts. The objectives of the Phase II investigation are to:

- characterize the soil stratigraphy;
- quantify VOCs which may be present in the soil at the plant; and
- evaluate the distribution of the VOCs in the soil

The scope of the proposed Phase II work will include:

- drilling of soil borings;
- collection of soil samples during drilling;
- preparation of well/boring log sheets;
- laboratory analysis of selected soil samples for U.S. EPA Method 8021;
- preparation of maps and cross-sections;
- preparation of isochemical contour maps, if appropriate; and
- identification of the absence or presence of potential source area(s).

The scope of work for the Phase II investigation including laboratory testing will be implemented by WWES' personnel in Grand Rapids, Michigan. All soil samples will be analyzed by WWES' Environmental Laboratory in Grand Rapids, Michigan.

#### 4.1 FIELD METHODS

The following is a summary of the field methodology to be implemented during the Phase II investigation. For a more detailed description of Environmental Laboratory Division protocol, refer to WWES's SOP's in Appendix C. The field work will be implemented in accordance to the Health & Safety Plan presented in Appendix B.

#### 4.1.1 SOIL BORINGS

The soil borings will be drilled using 4.25-inch inner diameter (ID) hollow-stem augers and a truck-mounted drill rig. The number, location, and depth of the soil borings will be determined based on the results of the Phase I investigation. The soil boring locations will be labeled with the prefix "SB".

Soil samples will be collected using a 1.5-inch ID split-barrel sampling device, 2 feet in length. The split-barrel sampling device will be driven into undisturbed sediments ahead of the lead auger. Soil samples will be collected every 2.5 feet to a depth of 10 feet and every 5 feet thereafter in accordance with ASTM Method D-1586. Upon completion of the soil borings, the boreholes will be backfilled with natural soil cuttings. Decontamination of the hollow stem augers and split-barrel sampling device is discussed in Section 4.3.2.

#### 4.1.2 SOIL SAMPLING

Two sets of soil samples will be collected from each sample interval. One set will be collected for chemical testing (see Table 1). This set of soil samples will be labeled with the soil boring identification followed by sample depth in parenthesis. The samples will be placed in an iced cooler chest for transport to the laboratory. The remaining amount of soil will be placed in a mason jar for field screening and visual inspection. A third set of soil samples will be collected as a duplicate set for laboratory analyses once every ten sample intervals. This sample set will be labeled with the soil boring identification and with the prefix "DP" followed by sample depth in parenthesis. One equipment rinse blank for every ten samples will also be submitted for laboratory testing (see Section 4.3.3).

#### 4.1.3 FIELD SCREENING

Field screening will be performed using a photoionization detector (PID) using a 11.7 eV lamp. The PID measures the total amount of volatile organics in the headspace of a sample in parts per million (ppm) with of precision of 0.1 ppm. The method of measuring the headspace of a soil sample is described in detail in Appendix C.

The field screening results will be recorded on well/boring log sheets along with a lithologic description of the sample and the number of blow counts required to advance the split-barrel sampler.

#### **4.1.4 SURVEY**

The location of the soil borings will be referenced with respect to the grid established during the Phase I investigation. The soil boring locations will be recorded to the 1.0-foot accuracy and will be plotted on the map created for the facility.

The ground elevation at the soil boring locations will be recorded to the 0.10-foot accuracy and referenced to the facility benchmarks established during the Phase I investigation.

#### 4.2 LABORATORY TESTING

The following is a summary of the laboratory methodology to be implemented during the Phase II investigation. For a more detailed description of laboratory protocol, refer to Appendix E.

The scope of analytical testing during the Phase II investigation includes laboratory analysis of soil samples, including duplicate samples. The number and identification of the soil samples submitted for chemical testing will be based on the Phase I results, the Phase II field screening results, and the location, depth, and soil type of individual samples. In addition, duplicate samples, equipment and trip blanks will be analyzed.

The soil samples, duplicate samples, equipment blanks, and trip blank sample, will be analyzed for purgeable and halogenated VOCs by gas chromatography (GC) using U.S. EPA Method No. 8021. A compound list and associated detection limits for Method No. 8021 for nonaqueous and aqueous matrices is presented in Tables 2 and 3, respectively. A list of required sample volumes, containers, preservation methods, and holding times for aqueous and nonaqueous matrices is included in Table 1.

#### 4.3 QA/QC PROGRAM

The QA/QC plan for the Phase II is based on WWES' standard laboratory protocol. The plan includes: 1) detailed field documentation; 2) decontamination of field equipment and proper sample handling; 3) laboratory analyses of duplicate samples, equipment blanks, and trip blanks; 4) proper sample handling and shipment; 5) stringent laboratory QA/QC procedures; and 6) the collection and laboratory analyses of duplicate samples.

#### 4.3.1 FIELD DOCUMENTATION

To facilitate accurate field records, various forms will be utilized by WWES field personnel to document routine procedures. Among these procedures, forms will be utilized for well/boring logs, sample identification tags, and chain of custody. Examples of these forms are included in Appendix D.

Logbooks will be used to record the data collection activities performed in the field. Entries in the field logbook will be described in as much detail as possible so that persons returning to the facility may reconstruct a particular situation without reliance upon memory. A guideline for taking field notes and records is presented in Appendix C.

Each entry in the logbook will be associated with a project-specific document number. All entries will be made in ink and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark, initialed and dated.

The title page of each logbook will contain the following:

- person to whom the logbook is assigned;
- logbook number;
- project name;
- project start and end dates.

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors, field sampling, or investigation team personnel and the purpose of their visit will also be recorded in the field logbook.

Whenever a sample is collected, or a measurement is made, a detailed description of the location of the station shall be recorded. The equipment used to collect samples will be listed, along with the time of sampling and the volume and number of containers collected for each sample interval. A sample identification number will be assigned prior to sample collection. The number of photographs taken of the station, if any, will be listed. All equipment used to make measurements will also be identified, along with the date of calibration. Any deviations to the proposed field activities will also be noted in the logbook. A guideline for taking field notes and records is in Appendix C.

#### 4.3.2 DECONTAMINATION OF FIELD EQUIPMENT AND SAMPLE HANDLING

All field equipment used during sample collection will be decontaminated prior to each use to reduce the likelihood of cross-contamination of soil samples. The drilling equipment will be steam cleaned or detergent scrubbed with a non-phosphate soap and rinsed with deionized water prior to use at each soil boring location. The sampling equipment (split-barrel sampler and stainless-steel spatula) will be washed with a non-phosphate soap and rinsed with deionized water prior to each sample interval.

Quality control provisions for soil boring sampling are as follows:

- Only "undisturbed" portions of the split-spoon core will be collected for sampling.
- The procedure for split-spoon sampling will consist of the method described in ASTM D 1586 found in Appendix C.
- Soil samples for volatile organic analyses will be collected as rapidly as is practical after the split-spoon sampler is opened. The sample will be placed directly into the sample vials with a stainless steel spatula.
- Soil samples will be collected in a jar for field screening with a PID or FID. These soil samples will be screened according to the technique described in the SOP entitled Jar Headspace Measurements in Unsaturated Soil Samples (Appendix C).
- Soil identification procedures will conform to WWES' standard soil classification found in Appendix C.

#### 4.3.3 DUPLICATE SAMPLES, EQUIPMENT BLANKS, AND TRIP BLANKS

Duplicate samples, equipment blanks, and trip blanks obtained will be analyzed during the Phase II investigation for VOCs (U.S. EPA Method No. 8021) to assess the quality of data resulting from the field sampling program.

Field duplicate samples will be collected and analyzed to check for sampling and analytical reproducibility.

Field equipment blanks will be collected to check for procedural contamination which may cause sample contamination. The general level of QC effort will be one equipment blank for every ten samples. The method of collection includes rinsing the split-barrel sampler with laboratory-grade deionized water and collecting the water in a volatile organic analysis (VOA) vial with no headspace.

Field trip blanks will be used to assess the potential for contamination of samples due to contamination migration during sample shipment and storage. One VOA trip blank consisting of laboratory-grade deionized water will be poured into the VOA vials with no headspace at the laboratory and included along with each cooler of samples. The trip blank will remain in the cooler during shipment from the laboratory to the facility, while it is stored at the facility (if necessary), and during shipment back to the laboratory. The trip blank will only be removed from the cooler for labeling.

#### 4.3.4 SAMPLE SHIPMENT

The sample shipment procedures summarized below will insure that the samples were collected properly and have arrived at the laboratory with the chain-of-custody intact. Examples of chain-of-custody forms, and other field and sampling activity forms are located in Appendix D.

Preservation techniques will be used to retard the chemical and biological changes that may take place after a sample is taken from its parent source. Samples collected in this sampling program will be placed in coolers and cooled with ice immediately upon collection and then transported to the WWES' Environmental Laboratory where they will be stored at 4°C until analysis. Table 1 lists sample preservation, holding time, and volume requirements for each type of sample collected during this phase of work.

The sample labeling and shipping procedures listed below will be implemented during the Phase II investigation.

- The field technician will be responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible will handle the samples.
- All bottles will be tagged with sample numbers and locations. An example of a sample tag is located in Appendix D.
- Sample tags will be completed for each sample using waterproof ink unless prohibited by weather conditions.
- Samples will be properly packaged for shipment and dispatched to the WWES' Environmental Laboratory for analysis, with a separate signed chain-of-custody record enclosed in each sample box or cooler identifying the contents. The original chain-of-custody record will accompany the shipment, and a copy will be retained by the sampler and returned to the WWES project manager.
- When transferring the possession of the samples, the individuals relinquishing and receiving the samples will sign, date, and note the time on the chain-of-custody record. This record documents transfer of custody of samples from the sampler to another person, to the analytical laboratory, or to/from a secure storage area. The chain-of-custody procedures for the WWES Environmental Laboratory are described in their Quality Assurance/Quality Control Procedures Manual (Appendix E).
- If the samples are sent by common carrier, a bill of lading shall be used. Receipts of bills of lading will be retained as part of the permanent documentation. If sent by

mail, the package will be registered with return receipt requested. Commercial carriers are not required to sign off on the custody form as long as the custody forms are sealed inside the sample cooler.

#### 4.3.5 LABORATORY QA/QC

All soil samples collected during field sampling activities at the facility will be analyzed by WWES' Environmental Laboratory in Grand Rapids, Michigan. The QA/QC Procedures Manual for the laboratory is presented in Appendix E.

Standard operating procedures for laboratory analyses are based on an analytical methods published by the U.S. EPA. For this project, U.S. EPA Method 8021 will be used. The standard operating procedure for this method, presented in Appendix F, specifies:

- procedures for sample preparation;
- instrument start up and performance check;
- initial and continuing calibration check requirements;
- specific methods for each sample matrix type; and
- required analysis procedures.

Calibration of laboratory equipment will be based on approved written procedures (see Appendix E). Records of calibration, repairs, or replacement will be filed and maintained by the designated laboratory analyst. These records will be filed at the location where the work is performed and will be subject to QA audit.

#### 4.4 REPORT

Upon completion of the drilling and sampling program, a report will be prepared by WWES summarizing the results of the soil survey and drilling and sampling programs. The data will be presented in tabular and graphic format. Isochemical contour maps and cross sections will be prepared, if appropriate.

The lateral and vertical extent of VOCs in the soil will be delineated. Identification of potential source areas will be made, if possible. Based on the results of the Phase I and II investigations, recommendations will be made regarding any appropriate remedial action. The draft report will be submitted to the Parties for their review and comment.

#### **REFERENCES**

- Einhorn, I.N., and others, 1991, Advances in Determining Soil and Ground Water Contaminants: Soils, p. 40-44.
- Imbrigiotta, T.E., and Martin, Angel, Jr., 1981. Hydrologic and chemical evaluation of the ground water resources of northwest Elkhart County, Indiana. U.S. Geological Survey: Water Resources Investigation 81-53, 140 p.
- Johnson, G.H., and Keller, S.J., 1972. Geologic map of the 1° and 2° Fort Wayne Quadrangle, Indiana, Ohio, and Michigan, showing bedrock and unconsolidated deposits: Indiana Department of Natural Resources, Geological Survey Division.
- Schneider, A.F., and Keller, S.J., 1970. Geologic map of the 1° and 2° Chicago Quadrangle, Indiana, Illinois, and Michigan, showing bedrock and unconsolidated deposits: Indiana Department of Natural Resources, Geological Survey Division.
- Weston-SPER, Technical Assistance Team, 1986. Regional ground water investigation of volatile organic contamination in Elkhart, Indiana. Technical report for U.S. Environmental Protection Agency, Region V, Contract No. 68-95-0017.

## TABLE 1

## U.S. EPA METHOD 8021 REQUIRED SAMPLE VOLUMES, CONTAINERS, PRESERVATION METHODS AND HOLDING TIMES FOR AQUEOUS AND NON-AQUEOUS MEDIA

## SELMER COMPANY ELKHART, INDIANA

| MATRIX      | CONTAINER           | PRESERVATION  | HOLDING TIME |
|-------------|---------------------|---------------|--------------|
| Non-Aqueous | 1-125 mL glass vial | cool 4°C; HCL | 14 days      |
| Aqueous     | 2-40 mL glass vials | cool 4°C; HCL | 14 days      |

## TABLE 2

## U.S. EPA METHOD 8021 FOR A NON-AQUEOUS MATRIX BY GC PARAMETERS AND ASSOCIATED DETECTION LIMITS

## SELMER COMPANY ELKHART, INDIANA

| COMPOUND                   | NON-AQUEOUS<br>DETECTION LIMITS<br>(mg/kg) |
|----------------------------|--|
| Bromodichloromethane       | 0.01                                       |
| Bromoform                  | 0.01                                       |
| Bromomethane               | 0.01                                       |
| Carbon tetrachloride       | 0.01                                       |
| Chlorobenzene              | 0.01                                       |
| Chloroethane               | 0.01                                       |
| 2-Chloroethylvinyl ether   | 0.01                                       |
| Chloroform                 | 0.01                                       |
| Dibromochloromethane       | 0.01                                       |
| 1,1-dichloroethane         | 0.01                                       |
| 1,2-dichloroethane         | 0.01                                       |
| 1,1-dichloroethene         | 0.01                                       |
| cis 1,2-dichloroethene     | 0.01                                       |
| 1,2-dichloroethene (total) | 0.01                                       |
| 1,2-dichloropropane        | 0.01                                       |
| cis-1,3-dichloropropene    | 0.01                                       |
| trans-1,3-dichloropropene  | 0.01                                       |
| Methylene chloride         | 0.01                                       |
| 1,1,2,2-tetrachloroethane  | 0.01                                       |
| Tetrachloroethene          | 0.01                                       |
| 1,1,1-trichloroethane      | 0.01                                       |
| 1,1,2-trichloroethane      | 0.01                                       |
| Trichloroethene            | 0.01                                       |
| Trichlorofluoromethane     | 0.01                                       |
| Vinyl chloride             | 0.01                                       |
| Benzene                    | 0.01                                       |
| Ethylbenzene               | 0.01                                       |
| Toluene                    | 0.01                                       |
| Xvlene                     | 0.01                                       |

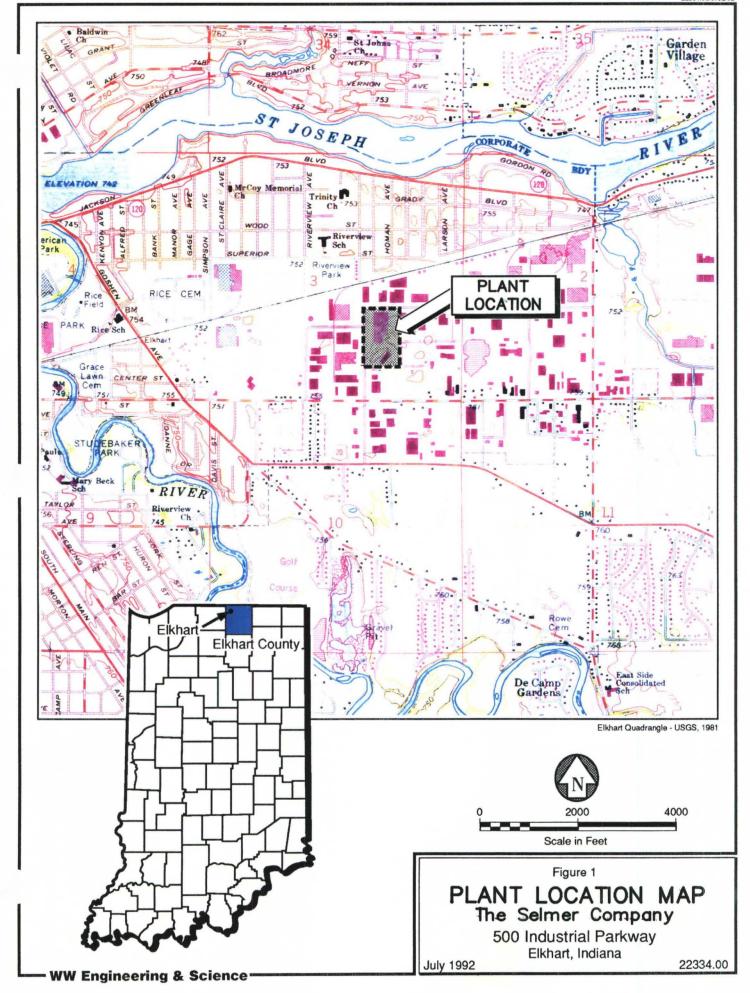
## TABLE 3

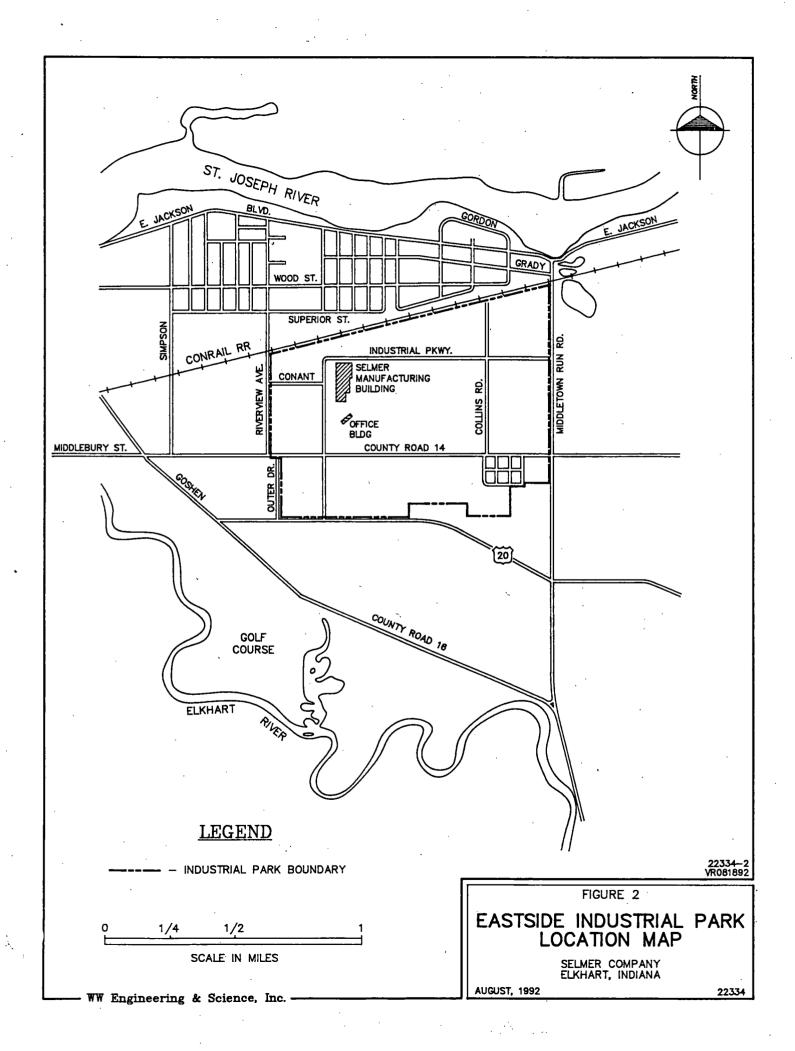
# U.S. EPA METHOD 8021 FOR AN AQUEOUS MATRIX BY GC PARAMETERS AND ASSOCIATED DETECTION LIMITS

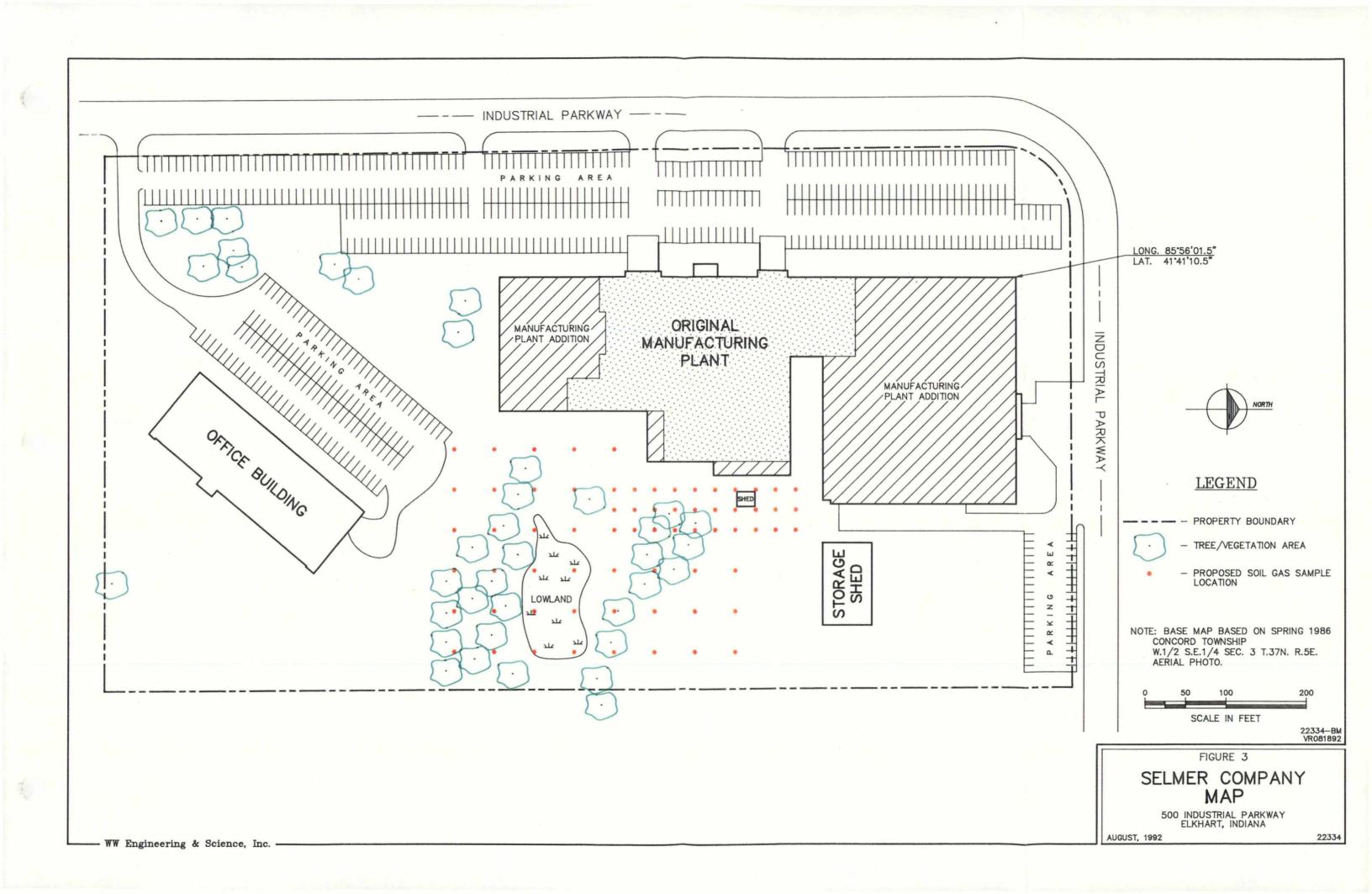
## SELMER COMPANY ELKHART, INDIANA

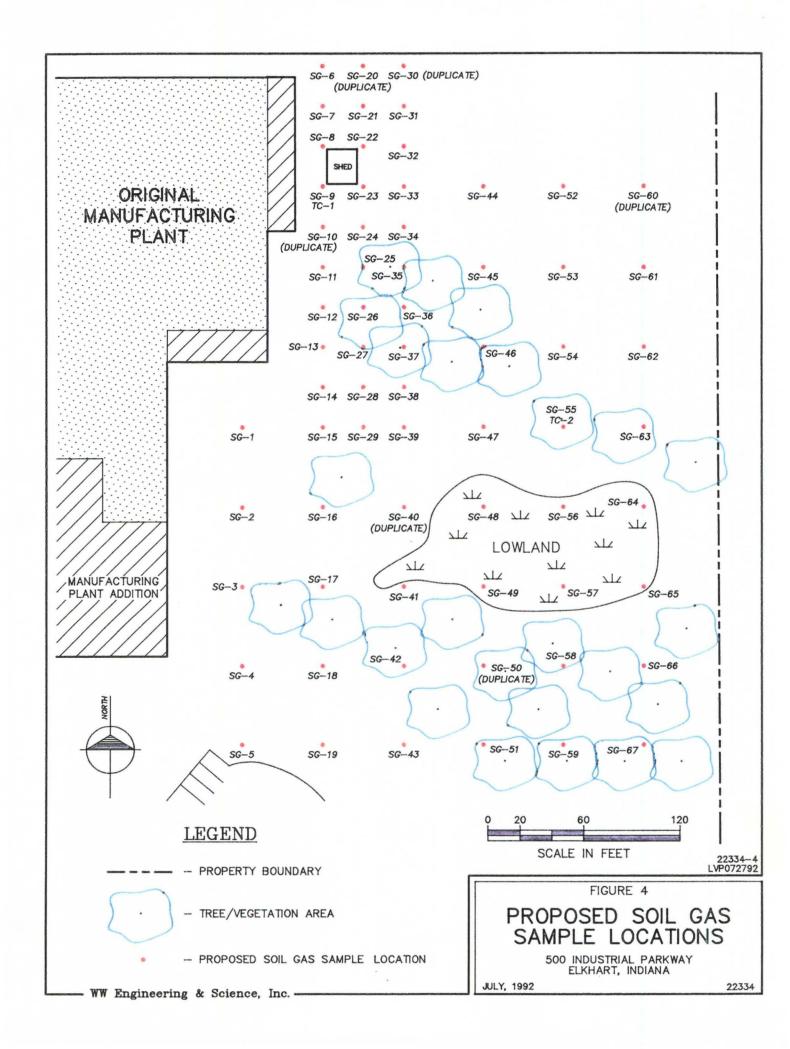
| COMPOUND                   | AQUEOUS<br>DETECTION<br>LIMIT (Mg/L) |
|----------------------------|--------------------------------------|
| Benzene                    | 0.001                                |
| Bromodichloromethane       | 0.001                                |
| Bromoform                  | 0.001                                |
| Bromomethane               | 0.001                                |
| Carbon tetrachloride       | 0.001                                |
| Chlorobenzene              | 0.001                                |
| Chloroethane               | 0.001                                |
| 2-Chloroethylvinyl ether   | 0.001                                |
| Chloroform                 | 0.001                                |
| Chloromethane              | 0.001                                |
| Dibromochloromethane       | 0.001                                |
| 1,2-Dichlorobenzene        | 0.001                                |
| 1,3-Dichlorobenzene        | 0.001                                |
| 1,4-Dichlorobenzene        | 0.001                                |
| Dichlorodifluromethane     | 0.001                                |
| 1,1-dichloroethane         | 0.001                                |
| 1,2-dichloroethane         | 0.001                                |
| 1,1-dichloroethene         | 0.001                                |
| trans-1,2-dichloroethylene | 0.001                                |
| 1,2-dichloropropane        | 0.001                                |
| cis-1,3-dichloropropene    | 0.001                                |
| trans-1,3-dichloropropene  | 0.001                                |
| Ethylbenzene               | 0.001                                |
| Methylene chloride         | 0.001                                |
| 1,1,2,2-tetrachloroethane  | 0.001                                |
| Tetrachloroethene          | 0.001                                |
| Toluene                    | 0.001                                |
| 1,1,1-trichloroethane      | 0.001                                |
| 1,1,2-trichloroethane      | 0.001                                |
| Trichloroethylene          | 0.001                                |
| Trichlorofluoromethane     | 0.001                                |
| Vinyl chloride             | 0.001                                |
| Xylene                     | 0.001                                |

## **FIGURES**









PETREX® Soil Gas Survey SOPs

STANDARD OPERATING PROCEDURES

FOR PETREX

ENVIRONMENTAL SOIL GAS SURVEYS

# STANDARD OPERATING PROCEDURES FOR PETREX ENVIRONMENTAL SOIL GAS SURVEYS

### 1.0 OPENING STATEMENT CONCERNING THE PURPOSE OF THIS DOCUMENT

The steps and information contained herein are the "Standard Procedures" for carrying out a PETREX soil gas survey. Minor alterations from these standard procedures may be implemented onsite by our field staff to adjust for unique survey conditions, such as frozen ground. The PETREX Technique is also used for oil and gas, geothermal, and mineral exploration; slight alterations in the operating procedures may be required for these specialized projects.

If any questions arise upon review of this document, please address your questions to NERI's technical staff. Please call:

Northeast Research Institute, Inc. (203) 677-9666 309 Farmington Avenue, Suite A-100, Farmington, Connecticut 06032

-or-

Northeast Research Institute, Inc. (303) 238-0090 605 Parfet Street, Suite 100, Lakewood, Colorado 80215

#### 2.0 SAMPLER PRODUCTION

# 2.1 Charcoal Bonding

PETREX collector wires are prepared by applying pre-sieved activated charcoal to the tips of ferromagnetic wires. Details of the procedure for preparing and bonding the activated charcoal are proprietary information. The resultant collector wires contain size-sorted activated charcoal bonded to the area within 1 cm of the tip. The specialty wires selected for this process have a Curie point of 358°C.

# 2.2 Sampler Tubes

Commercially available glass culture tubes, measuring 25 mm X 125 mm and having a screw cap closure, are prepared by washing in a biodegradable detergent, then rinsed in methanol and baked in an oven at  $180^{\circ}$ C for one hour.

# 2.3 <u>Cleaning of Collector Wires</u>

The charcoal bonded ferromagnetic wires are cleaned by heating in a special high vacuum apparatus at 358°C a total of 35 times. Wires are cleaned in lots of 32. From each lot, two wires are removed for immediate analysis by mass spectrometry to verify the cleanliness of the lot. The remaining 30 wires are then sealed in one clean culture tube under an inert atmosphere, assigned a lot number, and the lot(s) placed in inventory.

# 2.4 Lot Release and Repackaging

# 2.4.1 Quality Control and Quality Assurance

Prior to releasing inventory lots for a field survey, two collection wires from each lot are tested for cleanliness and adsorption potential. One wire is analyzed by mass spectrometry without exposure ("as is"), to verify that the lot is clean. The second wire is exposed to hexane vapor for two seconds, and then analyzed in order to verify that the charcoal is highly adsorptive.

# 2.4.2 Repacking for Shipment to the Field

Prior to shipment to the field, approved lots are removed from inventory, and the collector wires are repackaged in pre-cleaned sampler tubes under an inert atmosphere. From each lot containing 30 collector wires, 12 sampler tubes are packaged with 2 collector wires, and 2 sampler tubes with 3 collector wires. (The basis for having 2 wires in a tube is that it allows NERI to analyze one wire by standard Thermal Desorption-Mass Spectrometry (TD-MS), with the second wire being retained as a back-up or used later on for analysis by Thermal-Desorption-Gas Chromatography/Mass Spectrometry (TD-GC/MS). Sampler tubes containing 3 collector wires are used for the mass spectrometer set-up and gain adjustment procedure that is performed for each survey.)

# 2.5 <u>Custody Document</u>

A "custody document" accompanies each lot(s) of samplers released to the field for a project. This document accompanies the samplers through all transportation, field, and analysis steps.

### 3.0 FIELD OPERATIONS

# 3.1 <u>Locating Sampler Sites</u>

Sampler placement sites, usually predetermined on an accepted survey proposal, are located from a nearby, surveyable landmark using a compass and pacing, or some other measuring device (e.g., pacing wheel, hip chain, or tape measure). A transit may be used for more accurate placement, but such accuracy is seldom required.

# 3.2 Soil Coring

Once a sampler site has been established, a hole is cored to a predetermined depth; sampler placement depth is usually held constant for a given survey. A variety of tools is used for coring, depending on the nature of the surface cover and soil types. The holes should be vertical and as free from debris as possible. For grass covered sites, a coring shovel or core tube with a sledge hammer is used. When a survey is performed in areas covered by asphalt or concrete, a generator-powered rotary hammer drill with a carbide-tipped bit is used to drill a 1-1/2 inch diameter hole in the cover.

Down hole tools are decontaminated between each boring by following the procedure outlined in Section 3.9.

# 3.3 Sampler Placement

Immediately after the hole is cored, a sampler tube is removed from the Ziploc bag and the bag is resealed. The cap is then removed from the tube, and the tube is placed vertically, open end down, into the hole. The hole is then backfilled with the soil core which was removed. The cap is placed in a clean Ziploc bag to be used again later during sampler retrieval.

Samplers placed under asphalt or concrete are treated the same as those in uncovered soil, except for modifications to permit easy retrieval. To allow retrieval of these samplers, a length of galvanized steel wire is twisted around the neck of the tube and run to the surface so that the tube may be recovered by pulling on the retrieval wire. An aluminum plug is then placed near the top of the hole, and the remainder of the hole is plugged with quick setting hydraulic cement.

# 3.4 Sampler Location Marking

Each sampler location position is flagged using pin flags, spray paint or ribbon flagging, then the location is marked and numbered on a base map. A field notebook is used to record the date, sampler number, sampler location description, soil type, and general observations.

# 3.5 Residence Time

"Time calibration" samplers are included as part of every survey. These samplers are placed in a areas ranging from known or suspected contamination to background. Sets are retrieved and analyzed at specified time intervals to determine the appropriate field residence time for the survey. "Travel blank" samplers are also included to monitor for potential contamination acquired during transit. These samplers are transported along with the survey samplers, but the blanks are not opened until they are analyzed in the laboratory.

# 3.6 <u>Survey Retrieval</u>

All samplers from a survey are retrieved when analysis of the time calibration samplers indicates that there has been sufficient loading of gases onto the charcoal absorbent. The steps in the retrieval process are as follows: (1) Soil is gently excavated until the tube is exposed. (2) A cap is taken from the sealed Ziploc bag. The Viton seal is checked to make sure it is seated inside the cap. (3) The sampler is removed from the hole, and any dirt that is on the threads of the tube is wiped off with a clean cloth. If the tube is broken or cracked, the collection wires are transferred to a new tube using forceps. (4) The tube is capped tightly, numbered (see Section 3.7), and placed in a Ziploc bag. (5) Bore holes are filled or patched as required. (6) Flagging material and any other debris are removed from the survey area.

# 3.7 <u>Sampler Numbering</u>

Each sampler is immediately numbered according to the scheme established in the field notes and on the base map. The location number is written on an adhesive label which is then applied to the tube cap. In practice, labels are normally pre-numbered before starting the survey retrieval process, to ensure that no two sampler locations have the same number.

## 3.8 Sampler Shipment

Once all samplers have been retrieved, they are sealed in Ziploc bags, wrapped in bubble packing material, and packed tightly in a box for shipment. (Packing materials such as Styrofoam, vermiculite, or newspaper can introduce contaminants, and therefore should not be used for packaging.) The samplers, field notes, base map, and chain-of-custody document are either hand-carried back to NERI's laboratory or shipped by overnight courier service.

# 3.9 Decontamination

All down-hole equipment and tool parts which contact contaminated soil are constructed of heavy gauge steel. These tools are decontaminated between use at each sampling location by rotation through a four step cleaning process. The steps are:

- 1. Immersion and vigorous scrubbing in a mild solution of laboratory grade detergent until all visual accumulations of soil are removed.
- 2. Thorough rinsing with potable water.
- 3. Spray rinsing with methyl alcohol.
- 4. Air drying.

All derived liquids (and sediment) are contained in dedicated disposable vessels.

# 4.0 SAMPLER ANALYSIS

# 4.1 Numbering Check

Upon receipt of the samplers in NERI's laboratory, the number on each tube is recorded and any missing or duplicated numbers are noted. A missing number generally indicates that the sampler could not be retrieved. Samplers with identical numbers generally cannot be used unless their true site location can be established.

# 4.2 Holding Time

Exposed PETREX soil gas collection wires contain a minute quantity of various volatile organic compounds sorbed onto activated charcoal; the protective glass tube is effectively sealed when the Viton-lined cap is seated properly. Maximum holding time is a function of both the chemical stability of the sorbed compounds, and the integrity of the seal on the tube.

It has been our experience that PETREX soil gas samplers that are properly packaged after retrieval from the field, and stored under environmentally controlled conditions, typically remain compositionally unchanged for at least four months. Even with this long term stability, it is NERI's practice to analyze all samplers within three weeks of retrieval from the field.

# 4.3 Instrumentation

Thermal desorption is accomplished using a Fisher radio frequency power supply and a Curie point pyrolyzer designed by NERI and Extrel. The mass spectrometer used is an Extrel Spectrel quadrupole mass spectrometer. The analysis is controlled and recorded by DEC PDP 11/23 microcomputer. Following the analysis, all data are collected and archived on a personal computer (PC). Data for all active jobs are stored on personal computers, as well as on floppy disks. Data for all completed jobs are stored on magnetic tape and floppy disks in perpetuity.

# 4.4 Calibration

An Extranuclear Quadrupole Spectrometer equipped with a Curie-point pyrolysis/thermal desorption inlet is used for sampler analysis. Mass assignment and resolution are manually adjusted using a Perfluorotributylamine (PFTBA) standard. A linear correction, based on the known spectrum of PFTBA, is calculated. This correction is applied to a second PFTBA spectrum. If correct mass (M/Z) values are obtained, the operator proceeds to the next turning step. If not, Step 1 is repeated until correct masses are obtained.

Peak intensity ratios are set from the major peaks in the PFTBA spectrum using the following values:

| Mass  | -          | Spectrum           |
|-------|------------|--------------------|
| (M/Z) |            | <u>Intensities</u> |
|       |            |                    |
| 69    | =          | 100%               |
| 131   | <b>=</b> · | 25% <u>+</u> 5%    |
| 219   | =          | 35% <u>+</u> 5%    |
| 502   | ==         | 5% <u>+</u> 2%     |

The ion signal for mass (M/Z) 69 of PFTBA is measured at a preset sample pressure and detector voltage and compared to previous values at the same setting.

Electron energy is set to 70 electron volts and emission is set at 12 milliseconds. All other operating parameters, such as scans, scan range, mass offset are established in the computer program. These values may only be changed by the laboratory manager.

Tuning is performed at the beginning of a run, so that a complete survey is analyzed using the same instrument settings. Samplers are analyzed in random order.

# 4.5 Instrument Parameters

The instrument is operated with the following parameters:

Vacuum - < 3 X 10<sup>-6</sup> torr

Ionization Energy - 70.0 eV

Ionization Current - 12.0 mA

Desorption Time - 5.0 sec

Desorption Temperature - 358°C

Number of Scans/Sample - 30

Scan Rate - 1,250 amu/sec

# 4.6 <u>Mass Spectrometer Analysis and QA/QC</u>

Survey samplers are analyzed in random order. All samplers from one survey are analyzed without interruption from other projects.

The organic gases adsorbed onto the charcoal are thermally desorbed, separated according to ion mass, counted, and a mass spectrum of masses from 29 to 240 is obtained.

Periodic (approximately every 20 samples) machine background analyses are performed as a QC measure to assure minimal influence from internal communication. If there are peaks that are not related to atmospheric gases, the supervisor is notified and the mass spectrometer is shut down and cleaned as necessary.

A written sample number record is kept during the analysis to prevent accidental cross numbering. The mass spectrometer control program prompts the operator with a warning if a sample number is entered that has already been used. The operator then checks the current number, along with the disk storage location of the previously entered number, to resolve the true numbering situation.

# 4.7 <u>Data Filing</u>

The raw data file generated by each analysis is given a unique file name for storage.

## 4.8 Maintenance

# Frequency Activity

1,000 Analyses Cleaning of sample introduction area, ion source, and expansion chamber by in-house technicians.

4,000 Analyses Above noted procedures plus cleaning of lenses and quadrupoles.

Annually: Preventative maintenance program conducted by manufacturer's service representative.

# 5.0 DATA INTERPRETATION AND PRESENTATION

# 5.1 Compound Identification

Individual compounds are identified by comparing the mass spectrum that is obtained from each analysis to a library of reference mass spectra. Several thousand pure compound spectra have been developed by the Bureau of Standards and are available for spectral comparison. NERI has also developed its own library of spectra through headspace analysis of pure compounds using the PETREX process. Once a compound has been identified in this manner, the ion current (or ion count) of this compound is defined as the total ion current for the "parent peak" or the least interfered with peak of that compound. In a typical PETREX survey, numerous compounds are identified from each analysis.

# 5.2 <u>Compound Mapping</u>

# 5.2.1 Production of Sampler Location Map

Sampler location maps are created by placing the field base map on a digitizing board and entering each sampler location (and its respective number) as an X-Y coordinate relative to an origin. Alternatively, base maps may be supplied by the client in various CAD output formats on a floppy disk. Cultural and topographic features can also be digitized onto the map as reference points. The relative ion current (or ion count) for each compound can then be plotted at the exact sampler locations.

### 5.2.2 Production of PEIREX Isopleth Maps

The process of plotting total ion counts of indicator peaks from the compound(s) identified in the soil gas survey is computerized. Thus the summed ion counts from indicator peaks of identified compound(s) are matched with the sampler location on the base map, and the numeric value is plotted. The data are then contoured to take into account all other available data, such as geologic setting, soil types, groundwater conditions, type of contaminant, and site history.

The resultant maps show, per compound or class of compounds, isopleth lines that define the relative intensity of the signal throughout the survey area. Soil gas isopleth maps are useful for interpreting the areal extent of contamination, the location of source areas and relative "hot spots", and the direction of movement of the contaminants.

The entire PETREX process permits the collection, identification and mapping of numerous compounds simultaneously. This information is used to differentiate multiple compounds and multiple source areas within a single survey.

# 5.3 <u>Guidance on the Interpretation of Soil Gas Results</u>

Confirmation and quantification of soil gas results are generally conducted using standard field sampling methods for soil and groundwater analysis. The soil gas maps are used to guide the placement of borings and wells.

In general, extreme caution needs to be exercised when trying to extrapolate soil gas results (without the above sampling and analysis) to predict exact source of the soil gas signal (i.e. soil or groundwater), the depth of the signal, or concentrations of contaminants. In NERI's experience, the following hold true:

Results from soil gas surveys that have been conducted at a uniform shallow depth <u>cannot</u> be used to calculate the depth to the source or the concentration of contaminants at depth. Depth profiling (see Section 5.5.2) can greatly enhance the interpretation of the survey results.

Ion counts for any compound at one sample location can only be compared to another location within the same survey for the same compound. Ion counts of different compounds cannot be compared to each other. Also, the isopleth maps from one survey cannot be quantitatively compared to the results of any other survey, or between two surveys conducted at the same site at different times of the year. However, the same "hot spots" and migration pathways normally are detected in the same place over multiple surveys at a given site, allowing for migration.

### 5.4 Data Presentation

Once the data have been compiled, interpreted, and mapped, a report is produced for the client's use. Also, isopleth maps are finalized and printed using a sophisticated plotter and CAD software. These reports and maps are for the client's use only, and no report or map is released to anyone else without prior written consent of the client.

# 6.0 Additional Uses of PETREX SAMPLERS

PETREX samplers have numerous other uses, and the techniques described below are often incorporated into the soil gas survey design. (Specific instructions on sampling, shipment methods, and blanks are provided for each project.)

# 6.1 Headspace Analysis of Soils and Water

Headspace analysis can be used to establish a mass spectrometric pattern of compounds from soils or water; this pattern can then be used during interpretation of the soil gas survey by searching for the headspace pattern in the results obtained from the soil gas survey. This approach is very helpful for verifying sources or for mapping specific blends of commercial products at a site.

A soil sample is headspaced by collecting approximately 25 grams of soil in a thermochemically cleaned headspace container. A clean PETREX culture tube is often used. The sample is shipped to NERI's laboratory, where several PETREX collection wires are added. The sample is allowed to equilibrate for up to 24 hours, depending on the level of contamination. The exposed wires are then removed and prepared for thermal desorption mass spectrometric analysis as described earlier. A similar process is used for screening water samples.

# 6.2 <u>Depth Profiling</u>

In order to determine if the source of the soil gas signal is near surface or in a deeper vadose/saturated zone, depth profiling can be used. At each selected location, shallow bore holes are drilled a few feet apart to depths such as 1, 2, 4, and 6 feet deep. After all the loose cuttings and cavings have been removed from the bottom of the hole, a core of soil may be taken for headspace analysis. Next, a PETREX Sampler is installed as described earlier. The samplers remain in place for the same length of time as the rest of the PETREX survey.

Each of the PETREX sampling methods addresses different questions concerning the source of the VOC signal as detected during a soil gas survey.

In the case of soil headspace analysis, detection of VOCs indicates that the VOCs are actually contained within the soil matrix. When the VOC is anthropogenic in nature, the VOC presence is indicative of soil contamination at that depth interval.

When performing passive soil gas sampling with PETREX samplers, the sampler serves as both an extended headspace sampler relative to the soil matrix in its immediate vicinity, as well as measuring the relative rate of soil gas movement through that zone during the exposure period.

Soil gas movement through the vadose zone is theorized to be a diffusion process. If the soil headspace data indicate that the VOC is not present in the soil matrix, then the depth profiling samplers should show a relative increase of ion counts as the depth increases. By combining results from depth profiling and headspace analyses, the nature of the VOC source (near surface or deep vadose/saturated) can be inferred.

# Appendix B

Health and Safety Plan

# HEALTH AND SAFETY PLAN in support of the PHASE I SOIL GAS SURVEY PROGRAM and PHASE II DRILLING AND SAMPLING PROGRAM

at
THE SELMER COMPANY
500 INDUSTRIAL PARKWAY
ELKHART, INDIANA

for

NORTH AMERICAN PHILLIPS CORPORATION
THE SELMER COMPANY
and
MACMILLIAN, INC.

prepared by

WW ENGINEERING & SCIENCE 5555 GLENWOOD HILLS PARKWAY SE GRAND RAPIDS, MI 49588-0874

**AUGUST 1992** 

PROJECT NO. 22334

# 1.0 INTRODUCTION

This document describes the health and safety guidelines and procedures developed for the field activities associated with the Phase I Soil Gas Survey Program and the Phase II Drilling and Sampling Program for the Selmer Company facility, hereinafter referred to as the "facility". The facility is located in Elkhart, Indiana. The guidelines and procedures contained herein are based on the best available information at the time of this plan's preparation. Specific requirements will be revised when and if new information is received or conditions change significantly from original indications. Written amendments will document all changes made to this plan and all such amendments will be included in Attachment A. Where appropriate, specific Occupational Safety and Health Administration (OSHA) standards or other authoritative guidance will be cited and applied. All work will be coordinated through the Project Manager and will be performed in accordance with the provisions, guidelines, and procedures of this Facility Health and Safety Plan (SH&SP) and the requirements of OSHA's Hazardous Waste Operations and Emergency Response (HAZWOPER) standard, 29 CFR 1910.120. FAILURE TO COMPLY WITH THESE REQUIREMENTS MAY RESULT IN DISMISSAL FROM THE FACILITY.

# 1.1 FACILITY DESCRIPTION AND HISTORY

The facility consists of approximately 18.5 acres of lightly wooded land located in the west half of the southeast quarter of Section 3 of T37N, R5E of the Elkhart Quadrangle (see Figure 1). The main manufacturing building and associated storage buildings are located in the northern portion of the facility as shown in Figure 2. An office building with a parking lot is located at the south end of the facility. An asphalt parking area also exists along the western edge of the facility. There are no known underground storage tanks on the plant property. Topographic depressions located east of the manufacturing building and west of the office building periodically appear swampy due to surface water drainage.

The plant has been used exclusively since 1965 for the manufacture of brass musical instruments. The manufacturing process includes the use of trichloroethylene (TCE) as a degreaser. Vapor phase degreasers (VPD's) connected to a solvent recovery still have been used at the facility. Laboratory analyses of ground water samples from the eastern Elkhart area indicate the presence of volatile organic compounds (VOC's) including TCE, TCA, and methylene chloride, with TCE being the principal VOC detected in the area. The source, or sources, of the detected VOC's has not been identified to date.

### 1.2 SCOPE OF WORK

The field investigation activities at the facility will consist of the following major tasks, which are described in greater detail in the Field Investigation Work Plan:

Phase I. Soil Gas Survey Program

Task 1 - Survey the property and establish the soil gas grid.

Task 2 - Activate, install, and remove PETREX soil gas samplers.

Phase II. Drilling and Sampling Program

Task 1 - Drill soil borings.

Task 2 - Collect split-spoon samples.

# 1.3 KEY PERSONNEL AND ROLES/RESPONSIBILITIES

Contractor WW Engineering & Science

(WWES)

5555 Glenwood Hills Parkway

SE

**Grand Rapids, MI 49588-0874** 

(616) 942-9600

WWES Project Manager Scott Dennis, Ext. 233

WWES Project Geologist Lauryl Lefebvre, Ext. 446

WWES Field Services Manager Mike Potter, Ext. 336

WWES Survey Manager Randy Kolehouse, Ext. 212

WWES Health & Safety Officer to be determined

WWES Corporate Safety Director Ted Cline, Ext. 294

(616) 874-6236

Facility Contact Bert Kurtz

(219) 522-1675

WWES' Project Manager is responsible for coordinating field activities including, but not necessarily limited to, contractor oversight, sample collection and transport, and associated activity documentation. The Project Manager will report directly to the client contact and will act as liaison for the client with the U.S. EPA. The Project Manager will endeavor to ensure that all field activities are conducted in accordance with the Work Plan and this SH&SP.

WWES' Health & Safety Officer (SH&SO) has the authority and responsibility to implement this SH&SP. The SH&SO will enforce and verify compliance with the requirements of this SH&SP, and has the authority to immediately halt on-facility field activities due to unsafe conditions and/or behavior not in compliance with this SH&SP.

The SH&SO will maintain logs of all on-site project personnel, general weather data, and air monitoring data, and will complete incident investigation reports as necessary for each day of field activities. The SH&SO will also conduct facility safety meetings and determine appropriate upgrades or downgrades in Levels of Protection in accordance with the requirements of this SH&SP. The SH&SO, or designated alternate, will be responsible for informing all personnel involved in on-site project related activities of the contents of this SH&SP and ensuring that each person signs the Health & Safety Plan Acknowledgment Form in Attachment Z. By so signing, individuals are recognizing the potential hazards on-site and the procedures required to control the hazards and minimize their potential adverse effects.

# 2.0 SAFETY AND HEALTH RISK ANALYSIS

# 2.1 RISK ANALYSIS VS FIELD TASK

| Task: Phase I Soil<br>Gas Survey | Potential Hazard                  | Precautions   |
|----------------------------------|-----------------------------------|---|
| 1. Survey                        | Contact with Contaminated Soils   | <ul> <li>Use all PPE specified in Section 4.0.</li> <li>Do not kneel or sit on ground.</li> <li>Follow decontaminant procedures specified in Section 8.0.</li> </ul>                        |
|                                  | Contact with Contaminated Liquids | <ul> <li>Use all PPE specified in Section 4.0.</li> <li>Avoid contact with standing water in "swampy" area.</li> <li>Follow decontamination procedures specified in Section 8.0.</li> </ul> |
|                                  | Cold Stress                       | <ul> <li>Limit exposure time to cold temperatures, rain and wind.</li> <li>Keep dry.</li> <li>Wear layers of clothing.</li> <li>See Attachment B, Cold Stress.</li> </ul>                   |
|                                  | Physical Injury                   | <ul> <li>Exercise caution in vicinity of moving traffic.</li> <li>Be alert for slip, trip, and fall hazards.</li> <li>Follow Standard Work Orders in Section 2.3.</li> </ul>                |

| Task: Phase I Soil<br>Gas Survey                 | Potential Hazard                | Precautions   |
|--|---------------------------------|---|
| 2. Activate, install and collect PETREX samples. | Inhalation                      | <ul><li>Monitor with PID.</li><li>Respiratory protection.</li></ul>                                       |
|  | Contact with Contaminated Soils | See reference above.  |
|  | Noise                           | Use hearing protection devices of<br>sufficient noise reduction rating<br>(NRR) while using hammer-drill. |

| Task: Phase I Soil<br>Gas Survey (con't)                    | Potential Hazard                  | Precautions   |
|---|-----------------------------------|---|
| 2. Activate, install and collect PETREX samples (continued) | Heat Stress                       | <ul> <li>Slightly increase salt consumption in diet; DO NOT USE SALT TABLETS.</li> <li>Increase number and/or duration of rest breaks.</li> <li>Increase water intake.</li> <li>Monitor pulse at start of rest period. Must remain &lt;110 bpm to resume work.</li> </ul> |
|   | Electrical                        | <ul> <li>Use GFCI's.</li> <li>Follow 29 CFR 1926.400, Subpart K.</li> <li>Do not drill while standing in water.</li> <li>Insulated gloves and boot covers.</li> <li>Double-insulated hammer-drill.</li> <li>Discontinue activities during electrical storms.</li> </ul>   |
|   | Contact with Contaminated Liquids | See reference above.  |
|   | Cold Stress Physical Injury Skin  | <ul> <li>See reference above.</li> <li>See reference above.</li> <li>Use all PPE specified in Section 4.0.</li> <li>Good personal hygiene.</li> </ul>   |

| Task: Phase II<br>Drilling/Sampling | Potential Hazard                | Precautions   |
|-------------------------------------|---------------------------------|---|
| 1. Drill soil borings.              | Inhalation.                     | See reference above.  |
|                                     | Contact with Contaminated Soils | See reference above.  |
|                                     | Noise                           | <ul> <li>See reference above.</li> <li>Maintain appropriate distance from source(s).</li> </ul> |
|                                     | Heat Stress                     | See reference above.  |

| Task: Phase II Drilling/Sampling (Cont'd) | Potential Hazard                        | Precautions  |
|---|---|--|
| Drill soil borings.     (continued)       | Contact with Contaminated Liquids       | See reference above  |
|   | Cold Stress                             | See reference above  |
|   | Collapsing of Structure on<br>Personnel | <ul> <li>Stay clear of pathway of drill rig<br/>mast being raised and lowered.</li> <li>Verify raised mast is locked in<br/>position.</li> </ul> |
|   | Overhead Power Lines                    | <ul> <li>LOOK UP!</li> <li>Verify clearance of at least 15 feet.</li> <li>Chock wheels.</li> <li>De-energize lines, if necessary.</li> </ul>     |
|   | Skin                                    | See reference above  |
|   | Ventilation                             | <ul><li> Monitor with PID.</li><li> Use fans/blowers, if necessary.</li></ul>  |
| 2. Collect split-spoon soil samples.      | Inhalation                              | See reference above.   |
|   | Contact with Contaminated Soils         | See reference above.   |
| •   | Noise                                   | See reference above.   |
|   | Heat Stress                             | See reference above.   |
|   | Contact with Contaminated Liquids       | See reference above.   |
| ·   | Cold Stress                             | See reference above.   |
|   | Physical Injury                         | See reference above.   |
|   | Skin                                    | See reference above.   |

# 2.2 HEALTH ANALYSIS - SEE ATTACHMENT D

# 2.3 SAFE STANDARD WORKING ORDERS

# 2.3.1 Personal Precautions

- Eating, drinking, chewing gum or tobacco, smoking, applying cosmetics, or any other practice which increases the probably of hand-to-mouth contacts and associated transfer and ingestion of materials is strictly prohibited in any area designated potentially contaminated.
- Hands and face shall be thoroughly washed upon leaving the work area.

- Whenever decontamination procedures for personal protective equipment (PPE) are in effect, the entire body shall be thoroughly washed as soon as possible after the PPE is removed.
- No facial hair which may interfere with a satisfactory fit of the face-to-mask seal is allowed on personnel required to wear respirators.
- Contact with suspected contaminated surfaces shall be avoided at all times. Whenever possible do not: walk through puddles or discolored soils; kneel on ground; lean, sit, or place equipment on ground.
- Medications and/or alcohol can potentiate the effects from exposure to hazardous chemicals. Prescribed drugs shall not be taken when the potential for absorption, inhalation or ingestion of hazardous substances exists, unless specifically approved in writing by a qualified physician. Alcoholic beverage intake should be minimized or avoided, and is prohibited during working hours.
- All personnel must be familiar with safe standard operating procedures and adhere to the requirements of this Health and Safety Plan.
- Contact lenses shall not be worn when the hazard of a splash exists.
- Personnel shall be made aware of signs and symptoms of exposure to hazardous materials on-site, and of heat and cold stress, and shall promptly report to the Health and Safety Officer should any such symptoms be experienced.
- Respirators, if required, shall be inspected for worn or deteriorated parts prior to donning and shall be thoroughly disinfected and cleaned after each day's use or more often if necessary. Cartridges for air purifying respirators shall be replaced at least daily.

#### 2.3.2 OPERATIONS

- All personnel going on-site must be adequately trained and thoroughly briefed on anticipated hazards, equipment to be used and/or worn, safety practices to be followed, emergency procedures and communications.
- Personnel on-site must operate under the "buddy-system" for all activities in potentially impacted or contaminated areas.
- Visual contact must be maintained between pairs on-site and safety personnel.
   Entry team members should remain close together to assist each other in the event of an emergency.

- Personnel must practice unfamiliar operations prior to performing the actual procedures.
- Warning signals for evacuation and emergency escape routes and procedures must be established and clearly delineated prior to initiating on-site activities.
- Wind indicators visible to all personnel shall be strategically located throughout the facility. Red or yellow flagging secured to vehicle antennae may be used as wind indicators.
- Equipment and personnel in potentially contaminated areas shall be minimized, consistent with safe and effective operations.
- Frequent and regular inspections of field operations and activities will be conducted to ensure compliance with the Health and Safety Plan. If any changes in operations or conditions occur, the Health and Safety Plan must be modified to reflect such changes prior to the continuation of operations and activities.
- Fire prevention and protection shall be in accordance with 29 CFR 1926.150 Subpart F.

# 3.0 EMPLOYEE EDUCATION AND TRAINING

All on-site personnel involved with project-related activities shall provide certification of successful completion of either 24 hours (surveyors) or 40 hours (drillers and sampling personnel) of initial health and safety training, and up-to-date 8-hour refresher training, in accordance with OSHA's HAZWOPER Standard, 29 CFR 1910.120. At the minimum the training must include:

- a review of applicable federal and state regulations;
- general safety rules and practices;
- basics of toxicology and associated physiology;
- hazardous materials classifications and characteristics;
- respiratory protective equipment and training;
- personal protective equipment including chemical protective clothing;
- work zones and decontamination procedures and layouts;
- fire and explosion prevention and protection;
- first aid/emergency response procedures;

- confined space entry;
- · heat stress, cold stress and physical hazards;
- · atmospheric monitoring equipment and procedures;
- U.S. EPA/USCG/NIOSH/OSHA levels of protection (i.e., A,B,C & D);
- medical surveillance programs;
- · drum handling and sampling procedures; and
- basics of radiation safety.

# 4.0 PERSONAL PROTECTIVE EQUIPMENT (PPE)

As indicated in Section 2.1: Risk Analysis vs Field Task and based on current information, it is anticipated that most, if not all, of the tasks associated with the field investigation activities will be conducted in Level D PPE. The Level D PPE ensemble shall include:

- hard hat
- safety glasses (or chemical goggles/face shield if a splash potential exists)
- steel-toed/steel-shank safety boots
- chemical protective gloves (neoprene or Nitrile) if skin contact hazard exists
- Tyvek coveralls (optional)
- disposable HAZMAT boot covers (optional)

The Health and Safety Officer, or designated alternate, will make a determination as to the appropriate level of protection for all tasks based on PID readings in the worker's breathing zone. If sustained readings (>15 minutes) are over 25 ppm, workers will upgrade to Level C. Should Level C PPE be required, the Level C PPE ensemble shall include:

- Level D PPE listed above, plus
- Tyvek coveralls and boot covers (mandatory)
- Full-face air purifying respirators equipped with combination HEPA/organic vapor cartridges.

All respiratory protective equipment must be NIOSH/MSHA-approved. Gloves and boot covers shall be duct-taped to the chemical-resistant clothing.

# 5.0 MEDICAL SURVEILLANCE PROGRAM

All on-site personnel involved with project-related activities shall have been examined by a qualified physician within the past 12 months and deemed medically fit to wear the required PPE under conditions likely to be encountered at this facility, including heat and cold stress, in accordance with 29 CFR 1910.120(f) and 29 CFR 1910.134.

# 6.0 AIR MONITORING PROGRAM

The air monitoring program at this facility will consist of the following:

Monitor worker's breathing zones at least every 30 minutes for all tasks other than surveying. If breathing zone readings exceed 25 ppm, workers will upgrade to Level C. If breathing zone readings exceed 1,000 ppm, workers will promptly leave the area in an orderly fashion.

The instrument shall be calibrated according to manufacturers' instructions and all calibration data shall be recorded in a dedicated log book.

# 7.0 FACILITY CONTROL

# 7.1 FACILITY ACCESS AND RECORDKEEPING

Access to the field investigation area will be restricted to authorized personnel only. A log will be maintained by the Health & Safety Officer of all personnel entering and exiting each specific area throughout the course of the project.

# 7.2 SAFETY MEETING

Prior to the onset of investigation activities a safety meeting will be held on-site by the Health & Safety Officer. All personnel will be given a copy of this Health & Safety Plan. The plan will be explained and all personnel will sign a statement indicating they have read and understood the plan and have agreed to comply with all the guidelines and requirements contained therein. A copy of the plan will remain at the facility for the duration of the field investigation activities.

# **8.0 WORK ZONES AND DECONTAMINATION**

Individual exclusion zones will be established around each field investigation area with a decontamination zone located immediately adjacent to each exclusion zone. The zones will be delineated by the use of markers and/or ribbons. Eating and drinking will only be allowed in specific designated areas. Smoking will not be allowed on the plant property.

All personnel and equipment present in an exclusion zone must undergo decontamination before relocation. Soapy water and water rinse will be used as decontamination solutions and all spent solutions will be collected and disposed of according to applicable regulations. All disposable PPE will be double-bagged and clearly marked for proper disposal. All tools will be wiped with rags, washed with soapy water and water rinsed prior to removal from the plant property. Large equipment will be thoroughly decontaminated (with a steam cleaner, if necessary) prior to leaving the plant property. All personnel should shower as soon as practical after leaving the plant property.

# 9.0 EMERGENCY INFORMATION

# 9.1 EMERGENCY EQUIPMENT

A general first aid kit, portable eye wash units and Type A/B/C fire extinguishers will be available on-site at all times for the duration of the project.

# 9.2 EMERGENCY PRECAUTIONS

Material safety data sheets (MSDS) and other informational sheets are provided in Attachment B for hazardous substances suspected to be present and/or encountered. All personnel shall review the information therein and be familiar with listed signs and symptoms of exposure, and precautions and controls to minimize any adverse health effects.

# 9.3 EVACUATION PROCEDURES

Evacuation procedures, routes and rendezvous points will be identified and discussed at the safety meeting. Upon hearing a predetermined audible signal, all work area personnel will cease operations and promptly report to the designated rendezvous point where the Health & Safety Officer will take a head count. Any missing personnel will be identified and a list of the same with last known location will be provided to the emergency rescue squad upon arrival to the facility.

### 9.4 EMERGENCY PHONE NUMBERS/ADDRESSES

Elkhart Fire Department: (219) 295-7350

Elkhart Police Department: (219) 295-7070

Ambulance/Rescue Squad: (219) 295-7350

Hospital:

Elkhart General Hospital

600 East Boulevard; Elkhart, IN 46514

(219) 294-2621

**Poison Control:** 

1-800-392-9097

**WWES Corporate Safety Director:** 

616-942-9600 (Ext. 294) (work)

Ted Cline

(home)

Nearest Hospitals: The route to the nearest hospital is provided in the map in Attachment E.

# FIRST-AID EMERGENCY PROCEDURES

First aid is the treatment given a victim prior to the arrival of professional medical assistance. Note: First aid in no way replaces the attention of a physician. If there is any question about the seriousness of an accident victim's injury, contact a doctor as soon as possible. Give the following information:

- 1. What has happened—and when
- 2. Where the victim is located
- 3. What first aid has been provided

While the following guidelines are not a substitute for first-aid training, they will help you provide first aid in six serious emergency situations.

# **BROKEN BONES**

Call for medical assistance. If a doctor or ambulance can arrive within a short time, make no attempt to move the victim unless absolutely necessary. Attempt to immobilize the injured limb to prevent further injury. If the victim must be moved, splint the injured part with any available rigid material long enough to reach above and below the break. Secure the splint above and below the break. Never attempt to set a broken bone—wait for a doctor. Watch for signs of shock and treat as discussed below.

If broken bone pierces the skin, serious injury may have taken place. Make no attempt to move the injured limb. Apply a pressure dressing to control bleeding as described in the next section. Expect shock and treat as described.

### BLEEDING

Call for medical assistance. If bleeding is severe, apply firm, steady pressure to the wound with layers of sterile gauze pads or bandages. If they aren't available, use any cloth. Don't remove this dressing. If the pad becomes saturated with blood, add more layers. Bandage the pads firmly in place. If no gauze or cloth is available, close the wound with your fingers, holding it closed. Keep the victim lying down until a physician arrives. Elevate the bleeding part to help control bleeding. Never use a tourniquet to control bleeding unless you are dealing with an amputated, crushed, or mangled limb. Use a tourniquet ONLY as a last-resort effort to save a victim's life, because applying a tourniquet improperly may result in loss of limb.

### **BURNS**

Minor Burns: Immerse burned part in clear, cold water or apply ice for pain relief. Bandage with sterile pad or clean cloth. If pain persists, apply mild burn ointment.

Severe Burns: Call for medical assistance. Take immediate steps to relieve pain, prevent infection, and treat victim for shock as described below. If burn was caused by fire, boiling liquid, or hot metal, do not strip away clothing over the affected area. Keep air away from burn by covering area loosely in place. Apply no grease or ointment. Keep victim lying down. If conscious, give victim plenty of water.

Chemical Burns: Flush burn with large amounts of water. Cover burn with cleanest cloth available, and have victim lie down until a doctor arrives. For chemical burns of the eye, flush with great amounts of water immediately, cover the eye, and rush the victim to the doctor.

# **POISONING**

Call a doctor or poison control center at once: If victim loses consciousness, give no other first aid. If breathing stops, start mouth-to-mouth resuscitation. Follow the instructions of the doctor or poison control center.

# SHOCK

After any injury, expect shock—a condition in which vital body functions are slowed down. The symptoms are: weakness; cold, pale, clammy skin with beads of perspiration on face and palms; rapid, weak pulse; chills, nausea; irregular breathing. Any or all of these symptoms may be evident.

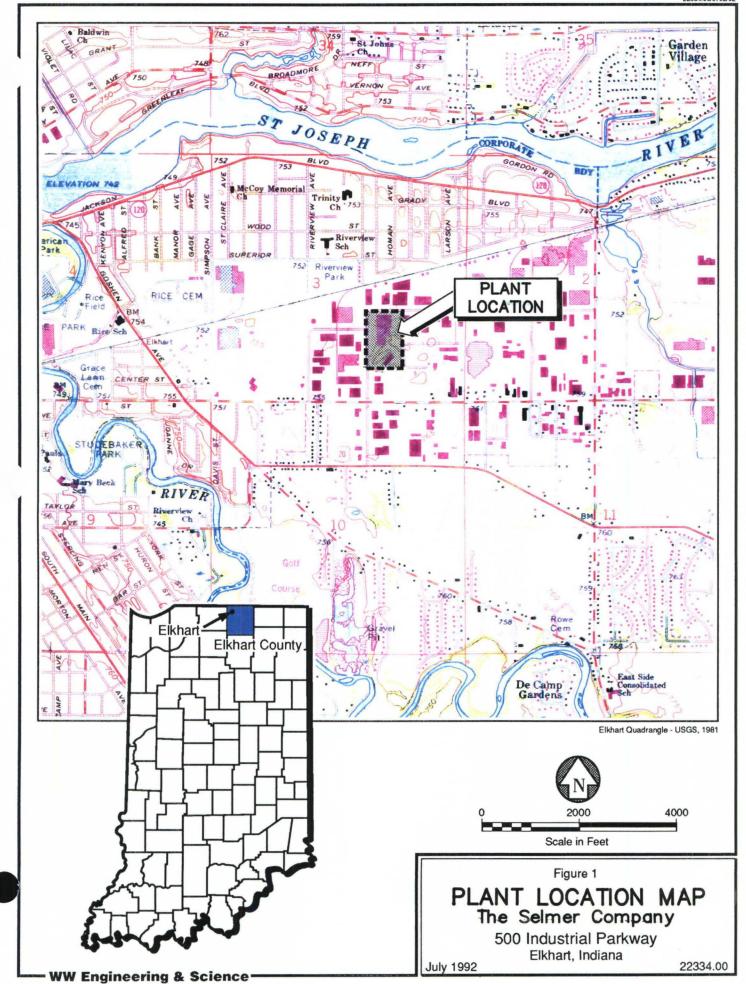
First aid involves keeping the victim warm—covered with blankets to prevent loss of body heat—and lying down. Keep victim's airway open. If victim vomits, turn his head to the side. If victim is conscious and able to swallow, give water. If victim becomes nauseated, stop liquids. Contact a doctor as soon as possible.

# BREATHING

If breathing stops for any reason, begin mouth-to-mouth resuscitation immediately. If possible, have someone else contact a doctor. Follow these steps:

- 1. Place victim on his or her back and determine if there is anything in the victim's mouth. If there is, turn the victim's head to one side and wipe out the mouth with a finger.
- 2. Straighten the victim's head and tilt it back so that the chin points up. Push the jaw down to keep the victim's tongue from blocking the airway.
- 3. Place your mouth over the victim's and pinch his nostrils shut with your fingers.
- 4. Breathe into the victim's mouth until the chest rises.
- 5. Remove your mouth and listen for the sound of escaping air. If you don't hear it, check the victim's head and jaw positioning and repeat the process. If there is no sound of escaping breath this time, turn the victim on his or her side and slap on the back between the shoulders. Check the mouth again for foreign matter.
- 6. Repeat steps 2, 3, and 4, removing your mouth to allow breath to escape from the victim's lungs. This process should be repeated about 12 times per minute for an adult. Above all, keep repeating the process until help arrives.

# **FIGURES**



#### TOLLING AGREEMENT

| This agreement ("Agreement) is entered into this day of           |
|---|
| , 1989, between the United States Environmental                   |
| Protection Agency (U.S. EPA) and Selmer Company. Selmer           |
| Company has been identified by U.S. EPA as a potentially          |
| responsible party in connection with two contaminated groundwater |
| plumes near its business site at 500 S. Industrial Parkway in     |
| Elkhart, Indiana. U.S. EPA and Selmer Company in consideration    |
| of the mutual covenants set out herein, agree as follows:         |

- 1. U.S. EPA contends that it presently has a cause of action against Selmer Company under Section 107 of the Comprehensive Environmental Response, Compensation, and Liability Act, 42 U.S.C. 9607, in connection with the incurrence of costs by U.S. EPA to provide safe drinking water to residences in a neighborhood generally bounded by Denver Street and Rice Street, Elkhart, Indiana.
- 2. U.S. EPA presently intends to file a complaint against Selmer Company on or before March 31, 1989, in the United States District Court.
- 3. U.S. EPA and Selmer Company are entering into this
  Agreement in order to allow groundwater flow information and other
  evidence to be gathered, pursuant to an Administrative Order by
  Consent, which may assist in a resolution of the claim alleged
  herein, without costly and protracted litigation.

- 4. This Agreement does not constitute in any way an admission of fact or liability by Selmer Company.
- 5. This Agreement does not constitute an admission or acknowledgement on the part of U.S. EPA of any applicable statute of limitations under the above cited statute, or any other applicable statutes or laws.
- 6. Selmer Company agrees that the time between April 1, 1989, and March 30, 1991, will not be included in computing the time limited by any statute of limitations under the causes of actions set forth in paragraph one hereto, if any statute of limitations is applicable. Nor will it be considered in a defense of laches or similar defenses concerning timeliness of commencing a civil action. Selmer Company shall not assert, plead or raise against U.S. EPA in any fashion, whether by answer, motion or otherwise, any defense or avoidance based on the running of any statute of limitations during the aforementioned period and any statute of limitations shall be tolled during and for this period.
- 7. U.S. EPA agrees not to institute the presently alleged cause of action set forth in paragraph one against the Selmer Company until the time period provided by the Administrative Order by Consent for gathering the evidence and information referred to in paragraph three has expired. The Administrative Order by Consent referred to herein and this instrument

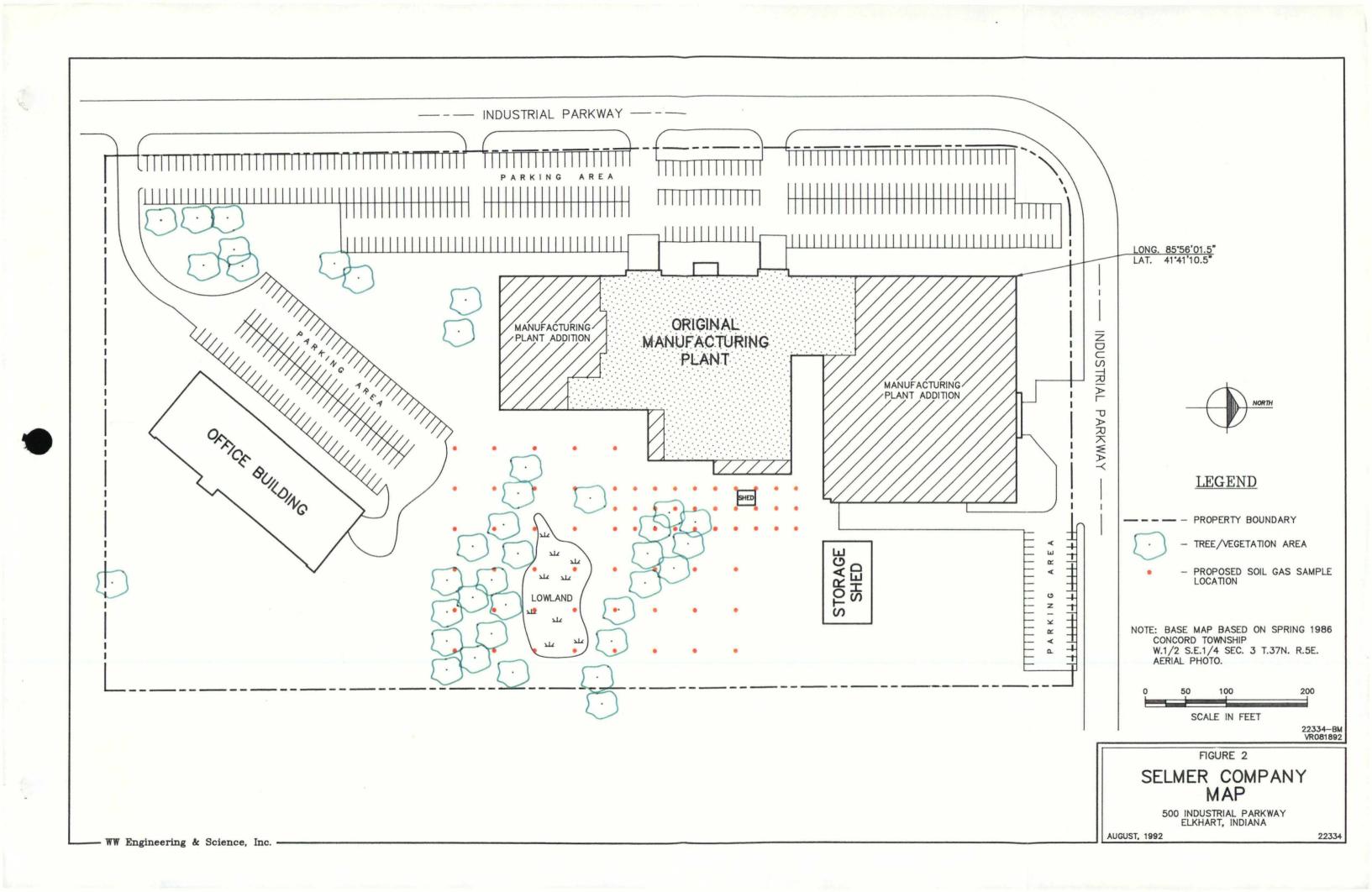
contain the entire agreement between the parties, and no statements, promises or inducements made by any party or agent of any party that is not contained in them shall be valid or binding; and they may not be enlarged, modified, or altered except in writing signed by the parties.

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## ATTACHMENTS

Health and Safety Plan Amendments

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## Attachment B

Cold Stress

## Supervisor's Safety Session

# **Safety in Winter**

Winter brings special health and safety risks, on and off the job. An extra hazard in generally warm areas is unreadiness to cope with sudden cold, ice, and snow.

## **Cold Stress**

Plan cold-weather jobs for the warmest part of the day—and include regular breaks in a warm spot, with an occasional warm drink (no alcohol or caffeine). Limit the time spent in the cold, and advise workers to keep moving. Smoking or bathing just before going out isn't a good idea, and alcohol is an obvious no-no. Hands, feet, face, and head should be covered outdoors. Suggest warm, loose, dry layers of clothing—cotton or wool inside and a waterproof outer layer. Garments that get wet should be exchanged promptly for dry ones.

## Skin Problems

Cold and wind, plus dry overheated rooms, make skin dry and chapped. This is uncomfortable and makes the skin more sensitive to other irritants, including chemicals. The best prevention is to cover as much skin as possible outdoors and wear protective clothing indoors on the job. Be sure workers wash thoroughly at the end of a shift and/or when removing protective clothing. It's also a good idea to use skin lotion after washing, and any time skin feels dry or itchy. Suggest adding moisture to the air at home with a humidifier, or even dishes of water on windowsills.

## Hypothermia

Extended exposure to cold can drive body temperature dangerously low (hypothermia). Wind, wet clothing, or physical exhaustion can cause hypothermia even when the thermometer is above freezing. It can lead to unconsciousness, occasionally death. At special risk are older people, or those who are overweight, smoke, drink, have poor circulation or allergies, or take various medications. Teach workers that feeling cold, pain in extremities, numbness, shivering, or drowsiness mean it's time to get warm and dry quickly.

### **Frostbite**

Direct exposure to cold or touching a subfreezing object can cause frostbite (frozen body tissue), though victims may be unaware of it. They feel cold, then numb, perhaps with tingling or pain. The skin turns white or grayish yellow, then reddish violet, then black, and may blister. The nose, cheeks, ears, fingers and toes are most vulnerable to frostbite, which can cause permanent tissue damage, with loss of movement in the affected parts—even unconsciousness or death from heart failure. Wrap

a frostbitten part in a sheet or blankets or warm it in warm (not hot) water. Don't rub, apply heat, or drink caffeine. Exercise a warmed body part, but don't walk on frostbitten feet.

### On the Road

Since driving is one of winter's greatest hazards, it would not be amiss to repeat some reminders on preparedness (e.g., stocking car with an ice scraper/ brush, snow shovel, and sand or other traction material; checking tire pressure, antifreeze, and windshield washer fluid) and extra caution on the road.

# Preparing for Winter A Safety Talk Outline

### **Prevent Cold Stress**

- Limit time in cold and keep moving. Take breaks where it's warm; have warm drinks (no alcohol/caffeine).
- Wear warm, loose, dry layers (cotton or wool inside, waterproof outside); protect hands, feet, face, head; change from wet to dry clothes immediately.
- No smoking, alcohol, bathing right before entering cold.

## **Skin Protection**

- · Cover skin outdoors to prevent dryness, chapping.
- Wear protective clothing on job to protect against irritants, and wash thoroughly at end of job
- Use lotions after washing and when skin feels dry/itchy.
- Use humidifier or containers of water to offset dryness at home.
   Hypothermia (less of body heat)
- Worst case: unconsciousness, occasionally death
- Risk conditions: extended exposure to cold, wind, wet clothes, tiring physical activity; (risk increased by age, overweight, poor circulation, smoking, alcohol, allergies, medications).
- · Symptoms: cold, pain, shivering, numbness, drowsiness.
- · Response: get warm and dry quickly.

## Frostbite (frozen body tissues)

- Worst case: tissue damage, loss of body-part movement, possible unconsciousness, death
- Risk conditions: exposure to cold, touching subfreezing object (nose, cheeks, ears, fingers, and toes most vulnerable).
- Symptoms: can include cold, numbness, possible tingling/pain; skin white or gray-yellow, then red-violet, then black; blisters
- Response: gently wrap in sheet or blankets or warm in warm (not hot) water; exercise warmed part (except feet); don't rub, apply heat, or drink caffeine.

### **Driving Safety**

- Keep ice scraper/snow brush, snow shovel, traction material (sand, etc.) in vehicle; clean snow, ice off vehicle before driving.
- Check tire pressure and be sure to have enough antifreeze, windshield washer fluid.
- · Reduce speed; stay farther behind other vehicle; watch for ice.

## Recognizing, Preventing and Treating Hypothermia

Man is a homoiotherm, a warm-blooded animal who must maintain a constant body heat of 98.6°F (37°C) so that the vital internal organs, particularly the heart and the brain, can perform properly. Workers in meat packing houses, freezer plants and cold storage facilities, outdoor construction workers, dockworkers, divers, firemen, fishermen and persons in like professions run the risk of endangering this delicate temperature balance by overexposure to cold conditions. Every day they face the possible danger of hypothermia.

Hypothermia is the cooling of the body core to a subnormal temperature caused by exposure to cold, wind and/or rain or by immersion in cold water. Under these conditions, which may easily occur when outside or water temperatures are well above freezing, the body begins to lose its essential heat more rapidly than it is able to

**Exposure Time Limits** Exposure Time Limit Remarks per & Mr. Work Parled 30 20 . providing that the worker is properly no limit clothed 10 0 alternating exposure — 1 hr. In and 1 hr. 4 hours out of cold area -20 - 30 reports differ, one recommends 15 mln. periods with a maximum of 4 per shift; two periods of another limits exposure to 1 hr. out of 30 min. each, every 4 hrs. with low chill factors, le\_no at least 4 hrs. wind; a third suggests that continuous apart - total operation for 3 hrs. at -53 has been exexposure- 1 hr. perienced with no tasting or harmful effects -70 -80 with completely enclosed headgear, heated air respirators and Insulated 5 minutes

Figure 1. Exposure time limits.

replace it. If such conditions are not soon reversed and the body core temperature is not returned to normal, the body's chemical reactions will be slowed and serious impairment or death may result.

### Variables

Hypothermia is usually due to a combination of two or more hazardous conditions. These conditions include the following variables:

Air Temperature: The cooling of the body core can occur at temperatures well above freezing. Caution should therefore be exercised to minimize the potential hazard. Cold exposure limits should be established according to Figure 1.

Water: Water conducts heat away from the body at a rate approximately 25 to 40 times faster than air temperatures. Such a rate applies not only to situations in which persons are totally immersed in water as a result of a dock accident etc., but also to situations in which perspiration, rain and mist are trapped against the body by clothing or footwear. Figure 2 depicts the maximum time limits for survival in cold water.

Wind: The body senses cold as a combination of both temperature and wind. The wind acts to increase the chilling effect of the temperature by blowing away the layer of insulation between the skin and the outside air. Thus, as the temperature falls, the wind has an increasingly greater effect on lowering the body's relative sense of cold. Figure 3 shows this cooling effect.

Clothing: Clothing provides insulation against the effects of cold, wind and rain by minimizing heat loss. It traps a layer of air between the warm body and the outside air, thus keeping in the body heat and keeping out the cold. The effectiveness of clothing is dependent upon the materials used to construct the clothing: for example. denim is loosewoven and easily penetrated by either air or water; duck and goosedown effectively prevent the wind from blowing away the warm air layer but are useless when wet (the insulating properties of cloth are severly reduced when wel); plastic and hylon afford good protection against the wind and the rain but offer little cold insulation. Consequently, the most effective way to dress is in layers of relatively light clothing, the uppermost of which should be waterproof and all of which can be easily donned or removed during the day to keep the body at a comfortable temperature.

Body Type: Survival time may be affected positively or negatively by body type. Body size, body attitude,



(Recognizing, Preventing and Treating Hypothermia - continued)

physical condition and amount of subcutaneous fat all contribute to the rate at which the body loses heat. The larger the body, the thicker the layer of insulating fat and the more stress it is able to withstand as a result of physical conditioning, the greater are the chances of survival in cold extremes.

Exposure: Even though the temperature of the hands and the feet can drop as much as 40° to 50° without permanent damage, it is important to keep them well covered because their high rate of heat loss may quickly affect the body core temperature. It is even more crucial to cover the head, because more than half the body's heat may be lost if the head is exposed. The sides of the chest and the groin area also have high heat loss rates, particularly in cold water exposures.

Fuel: If an individual becomes tired during the workday, he or she is more prone to heat loss. Therefore, the body must have a sufficient supply of fuel from which to

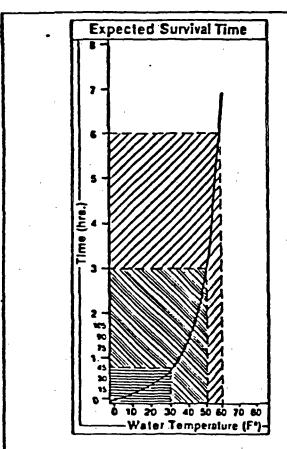


Figure 2. Maximum time limits for survival in cold water.

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Figure 3. Windchill factor.

draw energy. It is important to eat before going out into the cold and it is also important to carry a food supply (high-energy foods) if there is the possibility of extended cold exposure. Alcohol and drugs, which dilute the blood and numb the senses, lower the body's resistance to cold and consequently increase the risk of hypothermia.

Mental Attitude: Mental attitude plays an important role in any cold stress situation. Imperative to survival is a positive attitude including thought and preparation to prevent hypothermia, an understanding of the warning signs of hypothermia, knowledge of possible hazardous weather conditions and, if faced with an emergency, a clear head, a familiarity with first aid techniques and the will to survive.

### Preventative Measures

From the descriptions of the different variables which may contribute to hypothermia, it is easy to determine measures which, when implemented, can significantly reduce its incidence. Such measures include:

- · limiting exposure to cold temperatures, rain and wind
- keeping dry
- wearing several layers of clothing or insulated clothing, including a hat
- resting before any possible exposure to insure maximum strength
- eating well to maintain a high fuel level
- carrying extra high-energy foods
- carrying a first aid kit, including blankets
- using a thermal respirator when breathing cold air to prevent freezing the lung tissues
- · watching the weather
- thinking clearly
- maintaining a positive attitude in the event of an emergency
- being aware of the signs of hypothermia both in yourself and in others.

## Symptoms

When exposed to cold temperature and/or cold water, the body reacts instinctively in a pattern designed 10 preserve itself. It resorts to involuntary reactions originating in the brain. When the brain recognizes any the body to make adjustments to compensate for the imbalance. First, in an attempt to preserve normal temperatures in the vital internal organs, the blood vessels in the extremities constrict (vasoconstriction). This slows the blood flow to the arms and legs, preserving that energy and warm blood for the body core. If there is

inued heat loss and if the body core temperature ps below 95°F (35°C), the body then tries to generate more heat through shivering, which causes metabolic heat production to increase to several times the normal rate. This is the first real warning sign of hypothermia. Further heat loss, accompanied by a body core temperature drop to 90°F (32.2°C) or below, results in speech difficulty, loss of manual dexterity, slow reactions, mental confusion and muscle rigidity (muscle hypertonus). If exposure continues further until the body's resources are exhausted and if the cold blood reaches the heart and the brain, heart failure and coma will result and lead inevitably to death. Death occurs when the body core temperature falls below 78°F (25.6°C).

If exposure occurs in temperatures which are below freezing (30°F or below), frostbite or trench foot (immersion foot) may accompany or complicate the symptoms of hypothermia. Frostbite is the freezing of living tissues with a resultant breakdown of cell structure. Injury due to frostbite may range from superficial redness of the skin, slight numbness and blisters, to the obstruction of blood flow (ischemia), blood clots (thrombosis) or skin discoloration due to insufficient oxygen in the blood (cyanosis). Frostbite may occur if the skin comes into contact with objects whose surface temperature is below free-ing, such as metal tool handles. Trench foot is by continuous exposure to cold combined with ant dampness or immersion in water. Injuries in this case include permanent tissue damage due to oxygen

deficiency, damage to capillary walls, severe pain, blistering, tissue death and ulceration. Additionally, cold exposures may either induce or intensify vascular abnormalities. These include chilblain (a swelling or sore), Raynaud's disease, acrocyanosis (blueness of hands and feet) and thromboangiitis (inflammation of the innermost walls of blood vessels with accompanying clot formation). Workers suffering from these ailments should take particular prevautions to avoid chilling.

Hypothermia dumages both the body's internal temperature mechanisms (hypothalamus) and the peripheral mechanisms to prevent heat loss (vasoconstriction and perspiration). These effects may last up to three years.

## Trestment

If hypothermia occurs, certain first aid procedures can mean the difference between life and death for the victim. These include the following (as a general rule, treat all injuries in the order of their importance to preserving life):

## For Hypothermia:

- 1. Give artificial respiration and stop any bleeding, if necessary.
- 2. Bring the victim into a warm room or shelter as quickly as possible.
- 3. If the victim cannot be moved (spinal injury, etc.) carefully place newspapers, blankets or some other insulation between him and the ground.
- 4. Remove all wet clothing.
- 5. Provide an external heat source, for the body cannot generate its own heat. Wrap the victim in prewarmed blankets, place him or her in the liner of a portable hypothermia treatment unit, put the torso (not the extremities) into a tub of warm water or use body-to-body contact to rewarm the body core. These measures will slowly reopen the peripheral circula-

## Special Cold Water Survival Techniques

- 1. Always wear a personal flotation device, not only for its flotation characteristics, but also for the added insulation it will provide in the case of cold water immersion.
- Do not swim unless it is to a boat or a shore close by.
   The average swimmer can swim a distance of only 0.85 mile before being incapacitated by hypothermia, while wearing a personal flotation device.
- 3. Stay in or on a swamped boat.
- 4. Keep your head above water. Putting your head in the water increases heat loss.
- 5. Assume the HELP (Heat Escape Lessening Posture) or the Huddle position.
- 6. Maintain a positive attitude.



water, huddle so that the sides of the chests of different persons are

held close together. Remain still.

HUDDLE

## (Recognizing, Preventing and Treating Hypothermia - continued)

tion so as to minimize the possibility of after-shock or after-drop (the flowing of cooled, stagnated blood from the limbs to the heart), which may cause ventricular fibrillation, cardiac arrest or death.

- 6. Do not allow the victim to sleep.
- 7. Give warm, sweet drinks no alcohol or pain relievers.
- 8. Keep the victim still. Do not try to walk.

- 9. Do not rub numb skin.
- 10. Get medical help as soon as possible.

## For Frostbite:

- 1. Wrap the victim in woolen cloth and keep dry until he or she can be brought inside.
- 2. Do not rub, chase or manipulate frozen parts.
- 3. Bring the victim indoors.
- 4. Place the victim in warm water (102° to 105°F) and make sure it remains warm. Test the water by pouring it on the inner surface of your forearm. Never thaw affected parts if the victim has to go back out into the cold. The affected area may be refrozen.
- 5. Do not use hot water bottles or a heat lamp, and do not place the victim near a hot stove.
- 6. Do not allow the victim to walk if his or her feet are affected.
- 7. Have the victim gently exercise the affected parts once they are thawed.
- 8. Seek medical aid for thawing of serious forstbite, because the pain will be intense and tissue damage will be extensive.

## Attachment C

Heat Stress

# Heat Stress and Other Physiological Factors

Wearing PPE puts a hazardous waste worker at considerable risk of developing heat stress. This can result in health effects ranging from transient heat fatigue to serious illness or death. Heat stress is caused by a number of interacting factors, including environmental conditions, clothing, workload, and the individual characteristics of the worker. Because heat stress is probably one of the most common (and potentially serious) illnesses at hazardous waste sites, regular monitoring and other preventive precautions are vital.

Individuals vary in their susceptibility to heat stress. Factors that may predispose someone to heat stress include:

- · Lack of physical fitness.
- Lack of acclimatization.
- Age.
- Dehydration.
- Obesity.
- Alcohol and drug use.
- Infection.
- Sunburn.
- Diarrhea.
- Chronic disease.

Reduced work tolerance and the increased risk of excessive heat stress is directly influenced by the amount and type of PPE worn. PPE adds weight and bulk, severely reduces the body's access to normal heat exchange mechanisms (evaporation, convection, and radiation), and increases energy expenditure. Therefore, when selecting PPE, each item's benefit should be carefully evaluated in relation to its potential for increasing the risk of heat atress. Once PPE is selected, the safe duration of work/ test periods should be determined based on the:

- Anticipated work rate.
- Ambient temperature and other environmental factors.
- Type of protective ensemble.
- Individual worker characteristics and fitness.

## Monitoring

Because the incidence of heat stress depends on a variety of factors, all workers, even those not wearing protective equipment, should be monitored.

• For workers wearing permeable clothing (a.g., standard cotton or synthetic work clothes), follow recommendations for monitoring requirements and suggested work/rest schedules in the current American Conference of Governmental Industrial Hygienists' (ACGIH) Threshold Limit Values for Heat Stress [11]. If the actual clothing worn differs from the ACGIH standard ensemble in insulation value and/or wind and vapor permeability, change the monitoring requirements and work/rest schedules accordingly [12].



For workers wearing semipermeable or impermeable' encapsulating ensembles, the ACGIH
 standard cannot be used. For these situations, workers should be monitored when the temperature in the work area is above 70°F (21°C) [6].

## To monitor the worker, measure:

 Heart rate. Count the radial pulse during a 30-second period as early as possible in the rest period.

If the heart rate exceeds 110 beats per minute at the beginning of the rest period, shorten the next work cycle by one-third and keep the rest period the same

If the hart rate still exceeds 110 beats per minute at the next rest period, shorten the following work cycle by one-third [12].

 Oral temperature. Use a clinical thermometer (3 minutes under the tongue) or similar device to measure the oral temperature at the end of the work period (before drinking).

If oral temperature exceeds 99.6°F (37.6°C), shorten the next work cycle by one-third without changing the rest period.

If oral temperature still exceeds 99.6°F (37.8°C) at the beginning of the next rest period, shorten the following work cycle by one-third [12].

Do not permit a worker to wear a semipermeable or impermeable garment when his/her oral temperature exceeds 100.6 °F (38.1 °C)[12].

Body water loss, if possible. Measure weight on a scale accurate to ±0.25 lb at the beginning and end of each work day to see if enough fluids are being taken to prevent dehydration. Weights should be taken while the employee wears similar clothing or, ideally, is nude. The body water loss should not exceed 1.5 percent total body weight loss in a work day [12].

Initially, the frequency of physiological monitoring depends on the air temperature adjusted for solar radiation and the level of physical work (see Table 8-10). The length of the work cycle will be governed by the frequency of the required physiological monitoring.

### Prevention

Proper training and preventive measures will help evert serious illness and loss of work productivity. Preventing heat stress is particularly important because once someone suffers from heat stroke or heat exhaustion, that person may be predisposed to additional heat injuries. To avoid heat stress, management should take the following steps:

Adjust work schedules:

Modify work/rest schedules according to monitoring requirements.

Mandate work slowdowns as needed.

"Although no protective ensemble is "completely" impermeable, for practical purposes an outfit may be considered impermeable when calculating heat atress risk.

Rotate personnel: alternate job functions to minimize overstress or overexertion at one task.

Add additional personnel to work teams.

Perform work during cooler hours of the day if possible or at night if adequate lighting can be provided.

- Provide shelter (air-conditioned, if possible) or shaded areas to protect personnel during rest periods.
- Maintain workers' body fluids at normal levels. This is necessary to ensure that the cardiovascular system functions adequately. Daily fluid intake must approximately equal the amount of water lost in sweat, i.a., 8 fluid ounces (0.23 liters) of water must be ingested for approximately every 8 ounces (0.23 kg) of weight lost. The normal thirst mechanism is not sensitive enough to ensure that enough water will be drunk to replace lost sweat [14]. When heavy sweating occurs, encourage the worker to drink more. The following strategies may be useful:

Maintain water temperature at 50° to 60°F (10° to 15.6°C).

Provide small disposable cups that hold about 4 ounces (0.1 liter).

Have workers drink 16 ounces (0.5 liters) of fluid (preferably water or dilute drinks) before beginning work.

Urge workers to drink a cup or two every 15 to 20 minutes, or at each monitoring break. A total of 1 to 1.6 gallons (4 to 6 liters) of fluid per day are recommended, but more may be necessary to maintain body weight.

Weigh workers before and after work to determine if fluid replacement is adequate.

Encourage workers to maintain an optimal level of sphysical fitness;

Where indicated, acclimatize workers to site work conditions: temperature, protective clothing, and workload (see *Level of Acclimatization* at the end of this chapter).

Urge workers to maintain normal weight levels.

 Provide cooling devices to aid natural body heat exchange during prolonged work or severe heat exposure. Cooling devices include:

Field showers or hose-down areas to reduce body temperature and/or to cool off protective clothing. Cooling jackets; vests, or suits (see Table 8-5 for despita)

Train workers to recognize and treat hest stress.
 As part of training, identify the signs and symptoms of heat stress (see Table 8-11).

### Other Factors

PPE decreases worker performance as compared to an unequipped individual. The magnitude of this effect veries considerably, depending on both the individual and the PPE ensemble used. This section discusses the demonstrated physiological responses to PPE, the individual human characteristics that play a factor in these

Table 8-10. Suggested Frequency of Physiological Monitoring for Fit and Acclimatized Workers\*

| ADJUSTED TEMPERATURE               | NORMAL WORK ENSEMBLE           | MPERMEABLE ENSEMBLE            |
|------------------------------------|--------------------------------|--------------------------------|
| 90°F (32.2°C) or above             | After each 45 minutes of work  | After each 15 minutes of work  |
| 87.5°-90°F (30.8°-32.2°C)          | After each 60 minutes of work  | After each 30 minutes of work  |
| \$2.5°-87.5°F (28.1°-30.8°C)       | After each 80 minutes of work  | After each 60 minutes of work  |
| 77.5°-82.5°F (25.3°-28.1°C)        | After each 120 minutes of work | After each 90 minutes of work  |
| 72.5° - 77.5°F<br>(22.5° - 25.3°C) | After each 150 minutes of work | After each 120 minutes of work |

Source: Reference [13].

Table 8-11. Signs and Symptoms of Heat Stress\*

- Heat rash may result from continuous exposure to heat or humid air.
- Heat cramps are caused by heavy sweating with inadequate electrolyte replacement. Signs and symptoms include:
  - muscle spasms
  - pain in the hands, feet, and abdomen
- Heat exhaustion occurs from increased stress on various body organs including inadequate blood circulation due to cardiovascular insufficiency or dehydration. Signs and symptoms include:
  - pale, cool, moist skin
  - heavy sweating
  - dizziness
  - nausea
  - feinting
- Heat stroke is the most serious form of heat stress. Temperature regulation fails and the body temperature rises to critical levels.
   Immediate action must be taken to cool the body before serious injury and death occur. Competent medical help must be obtained. Signs and symptoms are:
  - red, hot, usually dry skin
  - lack of or reduced perspiration
  - nausea
  - dizziness and confusion
  - strong, rapid pulse
  - coma

responses, and some of the precautionary and training measures that need to be taken to avoid PPE-induced injury.

The physiological factors may affect worker ability to function using PPE include:

- Physical condition.
- Level of acclimatization.
- · Age.
- Gender.
- Weight.

### **Physical Condition**

Physical fitness is a major factor influencing a person's ability to perform work under heat stress. The more fit someone is, the more work they can safely perform. At a given level of work, a fit person, relative to an unfit person, will have [5,8,15,16]:

- Less physiological strain.
- A lower heart rate.
- A lower body temperature, which indicates less retained body heat (a rise in internal temperature precipitates heat injury).
- · A more efficient sweating mechanism.
- Slightly lower oxygen consumption.
- Slightly lower carbon dioxide production.

### Level of Acclimatization

The degree to which a worker's body has physiologically adjusted or acclimatized to working under hot conditions affects his or her ability to do work. Acclimatized Individuals generally have lower heart rates and body temperatures than unacclimatized individuals [17], and sweat sooner and more profusely. This enables them to maintain lower skin and body temperatures at a given level of environmental heat and work loads than unacclimatized workers [18]. Sweat composition also becomes more dilute with acclimatization, which reduces salt loss [8].

For work levels of 250 kilocalories/hour.

<sup>\*</sup>Calculate the adjusted air temperature (ta adj) by using this equation: ta adj \*F = ta \*F + (13 × % sunshine). Measure air temperature (ta) with a standard mercury-in-glass thermometer, with the bulb shielded from radiant heat. Estimate percent sunshine by judging what percent time the sun is not covered by clouds that are thick enough to produce a shadow. (100 percent sunshine = no cloud cover and a sharp, distinct shadow; 0 percent sunshine = no shadows.)

A normal work ensemble consists of cotton coveralls or other cotton clothing with long sleaves and pants.

<sup>\*</sup>Source. Referènce [6].



Acclimatization can occur after just a few days of exposure to a hot environment [15,16]. NIOSH recommends a progressive 6-day acclimatization period for the unacclimatized worker before allowing him/her to do full work on a hot job [16]. Under this regimen, the first day of work on site is begun using only 50 percent of the anticipated workload and exposure time, and 10 percent is added each day through day 6 [16]. With fit or trained individuals, the acclimatization period may be shortened 2 or 3 days. However, workers can lose acclimitization in a matter of days, and work regimens should be adjusted to account for this.

When enclosed in an impermeable suit, fit acclimatized individuals sweat more profusely than unfit or unacclimatized individuals and may therefore actually face a greater danger of heat exhaustion due to rapid dehydration. This can be prevented by consuming adequate quantities of water. See previous section on *Prevention* for additional information.

#### Age

Generally, maximum work capacity declines with increasing age, but this is not always the case. Active, well-conditioned seniors often have performance capabilities equal to or greater than young sedentary individuals. However, there is some evidence, indicated by lower sweat rates and higher body core temperatures, that older individuals are less effective in compensating for a given level of environmental heat and work loads [19]. At moderate thermal loads, however, the physiological responses of "young" and "old" are similar and performance is not affected [19].

Age should not be the sole criterion for judging whether or not an individual should be subjected to moderate heat stress. Fitness level is a more important factor.

#### Gender

The literature indicates that females tolerate heat stress at least as well as their male counterparts [20]. Generally, a female's work capacity averages 10 to 30 percent less than that of a male [8]. The primary reasons for this are the greater oxygen-carrying capacity and the stronger heart in the male [15]. However, a similar situation exists as with aging: not all males have greater work capacities than all females.

#### Weight

The ability of a body to dissipate heat depends on the ratio of its surface area to its mass (surface area/weight). Heat loss (dissipation) is a function of surface area and heat production is dependent on mass. Therefore, heat balance is described by the ratio of the two.

Since overweight individuals (those with a low ratio) produce more heat per unit of surface area than thin individuals (those with a high ratio), overweight individuals should be given special consideration in heat stress situations. However, when wearing impermeable clothing, that weight of an individual is not a critical factor in determining the ability to dissipate excess heat.

### References

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Material Safety Data Sheets
(MSDS)

Section 2.7 alth Analysis [For additional information refer to MSD]

ttachment D1

ge 1 of 1 **Chemical Properties** Chemical Name **IDLH** Route of Entry Exposure Limits **Symptoms** Proposed PEL 5,000 ppm Inhalation Fatigue, weakness; sleepiness; MW: 85 Methylene Chloride 8 hr TWA of lightheadedness BP: 104° F Sol: 2% CAS-RN: 75-09-2 25 ppm Ingestion Nausea F1 Pt: ? STEL 125 ppm Sp. Gr. 1.33 CH, CI, IP: 11.32eV Contact Irritates eves & skin VP: 350mmHg FRZ: -139 F [Carcinogen] UEL: 22% LEL: 14% 1,000 ppm Trichloroethylene Headache; vertigo; visual PEL 8 hr TWA Inhalation ' MW: 131 BP: 189° F disturbances; tremors; (trichloroethene) 50 ppm cardiac arrhythmia Sol: 0.1% Fl Pt: 90° CAS-RN: 79-01-6 STEL 200 ppm Nausea; vomiting IP: 9.45eV Sp. Gr. 1.46 Ingestion CIHC = CC1 2 [Carcinogen] VP: 58mmHg Irritates eyes & skin FRZ: -99°F Contact UEL: 10.5% LEL: 8% 1,000 ppm 1,1,1-trichlorethane PEL 8 hr TWA Inhalation Headache; lassitude; poor MW: 133 (methyl chloroform) equilibrium; CNS depression; BP: 165° F 350 ppm cardiac arrhythmia Sol: 0.4% CAS-RN: 71-55-6 STEL 450 ppm Fi Pt: None IP: 11.00eV Ingestion Sp. Gr. 1.34 Nausea VP: 100mmHg H<sub>3</sub> CCCl <sub>3</sub> Irritates eyes & skin FRZ: -23°F Contact UEL: 12.5% LEL: 7.5%



## ANALYTICAL STANDARD DATA SHEET

## **IDENTIFIERS**

Compound Name:

1,1,1-Trichloroethane

Synonyms:

Methyl chloroform

Chloroethene

CAS Number:

71-55-6

Molecular Formula:

C2H3Cl3

Molecular Veight:

133.4

Repository Number: EC-000010-01-XX

## STANDARD SOLUTION

Concentration:

 $1000 \pm 100 \,\mu g/mL*$ 

Solvent:

Methanol (Flammable)

Storage & Preservation: Store at ≤5°C. Transfer to tightly sealed vial with Teflon-lined septum or cap after opening.

equilibrate to room temperature before use.

### **PURITY**

Purity Assay of Neat Compounds:

QAR 98.1%

\*Concentration of the standard solution was corrected for purity at the time the solution was prepared.

## **HAZARDS**

This reference material is a dilute homogenous solution of 1,1,1-Trichloroethane in methanol. Hazard information for this specific solution is not available; therefore you must refer to the accompanying chemical profiles for both the solute and the solvent for applicable hazard data.

For comments or questions concerning these standards, please contact:

Quality Assurance Division

Environmental Monitoring Systems Laboratory

U.S. Environmental Protection Agency

Cincinnati, OH 45268

(513) 569-7325 or FTS 684-7325

MATERIAL SAFETY DATA SHEET

OH514370

OCCUPATIONAL HEALTH SERVICES, INC. 450 SEVENTH AVENUE, SUITE 2407

EMERGENCY CONTACT

JOHN S. BRANSFORD, JR. (615) 292-1180

NEW YORK, NEW YORK 10123 (800) 445-MSDS (212) 967-1100

) 967-1100

#### SUBSTANCE IDENTIFICATION .

CAS-NUMBER 71-55-6 RTEC-NUMBER KJ2975000

SUBSTANCE: METHYL CHLOROFORM

TRADE NAMES/SYNONYMS:

1,1,1-TRICHLOROETHANE: ALPHA-TRICHLOROETHANE: AEROTHENE TT:
METHYLTRICHLOROMETHANE: METHYLCHLOROFORM: TRICHLOROMETHYLMETHANE:
TRICHLOROETHANE: ETHANE, 1,1,1-TRICHLOROETHANE: CHLORTEN:
1,1,1-TRICHLORETHANE: TRICHLOROETHANE 111 DEGREASE COLD/VAPOR (ASHLAND):
ST-1000A CLEANER (STRESSCOAT): BLACO-THANE (BARON-BLANESLEE): PERM
ETHANE DG (DETREX CHEMICALS): SAFETY SOLVENT (LOCTITE CORPORATION):
STCC 4941176: RCRA U226: UN 2831: C2H3CL3: OHS14370

CHEMICAL FAMILY:
HALOGEN COMPOUND, ALIPHATIC

MOLECULAR FORMULA: C-H3-C-CL3

MOLECULAR WEIGHT: 133.40

CERCLA RATINGS (SCALE 0-3): HEALTH=2 FIRE=1 REACTIVIT:=0 FERSISTENCE=3
NFPA RATINGS (SCALE 0-4): HEALTH=2 FIRE=1 REACTIVITY=0

#### COMPONENTS AND CONTAMINANTS

COMPONENT: METHYL CHLOROFORM

PERCENT: 100.0

OTHER CONTAMINANTS: NONE .

EXPOSURE LIMIT:

METHYL CHLOROFORM (1,1,1-TRICHLOROETHANE): 350 PPM (1900 MG/M3) OSHA TWA; 450 PPM (2450 MG/M3) OSHA STEL 350 PPM (1900 MG/M3) ACGIH TWA; 450 PPM (2450 MG/M3) ACGIH STEL 350 PPM NIOSH RECOMMENDED 15 MINUTE CEILING

1000 POUNDS CERCLA SECTION 103 REPORTABLE QUANTITY
SUBJECT TO SARA SECTION 313 ANNUAL TOXIC CHEMICAL RELEASE REPORTING

#### PHYSICAL DATA

DESCRIPTION: CLEAR, COLORLESS LIQUID WITH A MILD CHLOROFORM-LIFE ODOR.

BOILING POINT: 165 F (74 C)

MELTING POINT: -26 F (-32 C)

SPECIFIC GRAVITY: 1.3390

EVAPORATION RATE: (BUTYL ACETATE=1)

SOLUBILITY IN WATER: 0.078% @ 25 C VAPOR DENSITY: 4.55

1,1,1-TRICHLOROETHANE

VAPOR PRESSURE: 100 MMHG @ 20 C

ODOR-THRESHOLD: 44-100.PPM

OTHER SOLVENTS (SOLVENT - SOLUBILITY):
SOLUBLE IN ACETONE, BENZENE, CHLOROFORM, METHANOL,
ETHANOL, CARBON DÍSULFIDE, ETHER, CARBON TETRACHLORIDE, N-HEPTANE.

ATHER PHYSICAL DATA VISCOSITY: 0.858 CPS 0 20 C

#### FIRE AND EXPLOSION DATA

FIRE AND EXPLOSION HAZARD SLIGHT FIRE HAZARD WHEN EXPOSED TO HEAT OR FLAME.

UPPER EXPLOSION LIMIT: 12:5%

LOWER EXPLOSION LIMIT: 7.5%

AUTOIGNITION TEMP.: 978 F (537 C)

FIRÉFIGHTING MEDIA: DRY CHEMICAL, CARBON DIOXIDE OR HALON (1987 EMERGENCY RESPONSE GUIDEBOOK, DOT P 5802.4).

FOR LARGER FIRES, USE WATER SPRAY, FOG OR STANDARD FOAM (1987 EMERGENCY RESPONSE GUIDEBOOK, DOT P 5800.4).

FIREFIGHTING:

STAY AWAY FROM STORAGE TANK ENDS. COOL CONTAINERS EXPOSED TO FLAMES WITH WATER FROM SIDE UNTIL WELL AFTER FIRE IS OUT (1987 EMERGENCY RESPONSE GUIDEBOOK, DOT P 5800.4, GUIDE PAGE 74).

EXTINGUISH USING AGENTS FOR SURROUNDING FIRE. COOL FIRE-EXPOSED CONTAINERS WITH FLOODING AMOUNTS OF WATER APPLIED FROM AS FAR A DISTANCE AS POSSIBLE. DO NOT ALLOW RUN-OFF WATER INTO SEWERS AND WATER SOURCES. AVOID BREATHING VAPORS.

#### TRANSPORTATION

DEPARTMENT OF TRANSPORTATION HAZARD CLASSIFICATION 49CFR172.101: ORM-A

DEPARTMENT OF TRANSPORTATION LABELING REQUIREMENTS 49CFR172.101 AND SUBPART E:

DEPARTMENT OF TRANSPORTATION PACKAGING REQUIREMENTS: 49CFR173.605 EXCEPTIONS: 49CFR173.505

#### TOXICITY

METHYL CHLOROFORM (1,1,1-TR[CHLOROETHANE):

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1,1,1-TRICHLOROETHANE

Page 1 of 7

IRRITATION DATA: 450 PPM/8 HOURS EYE-MAN; 5 GM/12 DAYS INTERMITTENT SKIN-RABBIT MILD: 20 MG/24 HOURS SKIN-RABBIT MODERATE; 100 MG EYE-RABBIT MILD; 2 MG/24 HOURS EYE-RABBIT SEVERE. TOXICITY DATA: 27 GM/M3/10 MINUTES INHALATION-MAN LCLO; 350 PPM INHALATION-MAN TCLO; 200 PPM/4 HOURS INHALATION-MAN TCLO; 920 PPM/70 MINUTES INHALATION-HUMAN TCLO: 18000 PPM/4 HOURS INHALATION-RAT LC50: 3911 PPM/2 HOURS INHALATION-MOUSE LC50: 24400 MG/M3 INHALATION-CAT LC50; 1 GM/KG SKIN-RABBIT LDLO; 670 MG/HG ORAL-HUMAN TOLO; 10300 MG/KG ORAL-RAT LD50; 11240 MG/KG CRAL-MOUSE LDSO: 5660 MG/KG CRAL-RABBIT LDSO: 9470 MG/KG ORAL-GUINEA PIG LD50; 750 MB/KG ORAL-DOG LD50; 16 GM/KG SUBCUTANEOUS-MOUSE LD50: 500 MG/KG SUBCUTANEOUS-RABBIT LDLO: 95 MB/KB INTRAVENOUS-DOG LDLO; 3593 MB/KB INTRAPERITONEAL-RAT LD50; 3636 MG/KG INTRAPERITONEAL-MOUSE LD50; 3100 MG/KG INTRAPERITONEAL-DOG LD50; 15800 MG/KG SKIN-RABBIT LD50 (EPA-600/8-82-003F, 1984); MUTAGENIC DATA (RTECS); REPRODUCTIVE EFFECTS DATA (RTECS). CARCINOGE! STATUS: ANIMAL INADEQUATE EVIDENCE (IARC CLASS-3). LOCAL EFFECTS: IRRITANT- INHALATION, SKIN, EYE. ACUTE TOXICITY LEVEL: SLIGHTLY TOXIC BY INHALATION, DERMAL ABSORPTION AND INGESTION. TARGET EFFECTS: CENTRAL NERVOUS SYSTEM DEPRESSANT. POISONING MAY ALSO AFFECT THE HEART AND POSSIBLY LIVER AND KIDNEYS. AT INCREASED RISK FROM EXPOSURE: PERSONS WITH PRE-EXISTING SKIN DISORDERS. LIVER DISEASE OR CARDIOVASCULAR DISEASE. ADDITICHAL DATA: ALCOHOL MAY FOTEHTIATE BOTH CARDIAC AND HEPATIC TOXICITY. EPINEPHRIME OR OTHER STIMULANTS MAY INDUCE VENTRICULAR ARRHYTHMIAS.

#### HEALTH EFFECTS AND FIRST ALD

INHALATION:

METHYL CHLOROFORM (1,1,1-TRICHLOROETHANE):

IRRITANT/MARCOTIC. 1000 PPM IMMEDIATELY DANGEROUS TO LIFE OR HEALTH.
ACUTE EXPOSURE- EXPOSURE TO 300 PPM FOR 60 MINUTES SHOULD CAUSE NO EFFECT
EXCEPT FOR A DISTINCTIVE ODOR WHILE 900-1000 PPM FOR 20 MINUTES MAY CAUSE
MILD RESPIRATORY TRACT IRRITATION AND PROMPT BUT MINIMAL IMPAIRMENT OF
EQUILIBRIUM WHICH MAY BE ACCOMPANIED BY HEADACHE, LASSITUDE AND ATAXIA.
IMPAIRED PERFORMANCE OF BEHAVIORAL TESTS WAS ALSO REPORTED AT 1000 PPM.
HIGHER LEVELS OF 2000-3000 PPM MAY CAUSE INCOORDINATION, AMESTHESIA,
LOSS OF CONSCIOUSNESS, COMA AND DEATH. EXCESSIVE CONCENTRATIONS OF
10,000 PPM MAY CAUSE DEATH DUE TO RESPIRATORY OR CARDIAC FAILURE.
CARDIAC SENSITIZATION MAY BE A CONTRIBUTING FACTOR. OTHER EFFECTS MAY
INCLUDE NAUSEA, VOMITING, DROWSINESS, CONVULSIONS, FALL OF BLOOD PRESSURE
LIVER AND KIDNEY DAMAGE, BRADYCARDIA AND BLOOD CLOTTING CHANGES.

CHRONIC EXPOSURE- NO ADVERSE EFFECTS RELATED TO EXPOSURE WERE REPORTED IN VOLUNTEERS EXPOSED TO 500 PPM FOR 7 HOURS A DAY FOR 5 DAYS, OR IN WORKERS EXPOSED TO 200 PPM FOR SEVERAL MONTHS TO 6 YEARS. EXPOSURE OF ANIMALS FOR 3 MONTHS AT CONCENTRATIONS FROM 1000 TO 10,000 PPM CAUSED SYMPTOMS OF CENTRAL NERVOUS SYSTEM DEPRESSION AND SOME PATHOLOGICAL CHANGES IN THE LIVERS AND LUNGS OF SOME SPECIES. REPRODUCTIVE EFFECTS HAVE BEEN REPORTED IN ANIMALS.

FIRST AID- REMOVE FROM EXPOSURE AREA TO FRESH AIR IMMEDIATELY. IF BREATHING HAS STOPPED, GIVE ARTIFICIAL RESPIRATION. MAINTAIN AIRWAY AND BLOOD PRESSURE AND ADMINISTER OXYGEN IF AVAILABLE. KEEP AFFECTED PERSON MARM AND AT REST. TREAT SYMPTOMATICALLY AND SUPPORTIVELY. ADMINISTRATION OF OXYGEN SHOULD BE PERFORMED BY QUAL:FIED PERSONNEL. GET MEDICAL ATTENTION IMMEDIATELY.

METHYL CHLOROFORM (1,1,1-TRICHLOROETHANE):
IRRITANT.

ACUTE EXPOSURE- DIRECT CONTACT MAY CAUSE IRRITATION AND REDNESS. VAPORS ARE POORLY ABSORBED, BUT THE LIQUID, ESPECIALLY IF CONFINED UNDER AN IMPERMEABLE BARRIER MAY BE ABSORBED TO SOME EXTENT. THIS ALONE IS UNLIKED TO RESULT IN TOXIC EFFECTS, BUT MAY ADD TO THE EFFECTS OF INHALATION EXPOSURE

CHRONIC EXPOSURE- REPEATED SKIN CONTACT MAY PRODUCE A DRY, SCALY, FISSURED DERMATITIS DUE TO THE DEFATTING PROPERTIES OF THE LIQUID, AND POSSIBLY BURNS.

FIRST AID- REMOVE CONTAMINATED CLOTHING AND SHOES IMMEDIATELY. WASH AFFECTED AREA WITH SOAP OR MILD DETERGENT AND LARGE AMOUNTS OF WATER UNTIL NO EVIDENCE OF CHEMICAL REMAINS (APPROXIMATELY 15-20 MINUTES). GET MEDICAL ATTENTION IMMEDIATELY.

EYE CONTACT:

SKIN CONTACT:

METHYL CHLOROFORM (1,1,1-TRICHLOROETHANE): 1881 TANT.

ACUTE EXPOSURE - EXPOSURE TO 500 PPM MAY CAUSE IRRITATION AND REDMESS.
DIRECT CONTACT WITH THE LIQUID MAY CAUSE TEMPORARY INJURY WITH COMPLETE
RECOVERY EXPECTED IN 48 HOURS. DIRECT APPLICATION TO THE EVES OF RABBITS
HAS CAUSED CONJUNCTIVAL IRRITATION, BUT NO CORNEAL DAMAGE.
CHRONIC EXPOSURE - REPEATED OR PROLONGED CONTACT MAY CAUSE CONJUNCTIVITIS.

FIRST AID- MASH EYES IMMEDIATELY WITH LARGE AMOUNTS OF WATER OR MORMAL SALIM OCCASIONALLY LIFTING UPPER AND LOWER LIDS, UNTIL NO EVIDENCE OF CHEMICAL REMAINS (APPROXIMATELY 15-20 MINUTES). GET MEDICAL ATTENTION IMMEDIATELY.

INGESTION.

METHYL CHLOROFORM (1,1,1-TRICHLOROETHANE): NARCOTIC.

ACUTE EXPOSURE- MAY CAUSE NAUSEA, VOMITING, DIARRHEA, GASTROINTESTINAL DISTURBANCES AND ABDOMINAL PAIN FOLLOWED BY CENTRAL NERVOUS SYSTEM DEPRESSION WITH HEADACHE, DIZZINESS, WEAKNESS, INCOORDINATION, MENTAL CONFUSION AND UNCONSCIOUSNESS. DEATH MAY OCCUR FROM CHRONIC RESPIRATORY FAILURE. OTHER SYMPTOMS AS DESCRIBED IN ACUTE INHALATION MAY ALSO OCCUR. MYOCARDIAL SENSITIZATION TO EPINEPHRINE AND SUBSEQUENT DEATH DUE TO CARDIAC ARREST MAY OCCUR. ASPIRATION MAY RESULT IN PULMONARY EDEMA OR CHEMICAL PNEUMONITIS.

CHRONIC EXPOSURE- REPRODUCTIVE EFFECTS HAVE BEEN REPORTED IN ANIMALS.

FIRST AID- TREAT SYMPTOMATICALLY AND SUPPORTIVELY. GET MEDICAL ATTENTION AND ADVICE ON WHETHER TO USE GASTRIC LAVAGE. EXTREME CARE MUST BE TAKEN TO PREVENT ASPIRATION. A CUFFED ENDOTRACHEAL TUBE USED BY DUALIFIED MEDICAL PERSONNEL MIGHT BE ADVISABLE. KEEP HEAD LOWER THAN HIPS TO PREVENT ASPIRATION SHOULD VOMITING OCCUR.

ANTIDOTE:

NO SPECIFIC ANTIDOTE. TREAT SYMPTOMATICALLY AND SUPPORTIVELY.

REACTIVITY SECTION

#### REACTIVITY: .

SLOWLY DECOMPOSES OVER TIME YIELDING HYDROGEN CHLORIDE. AN INHIBITOR MAY BE ADDED TO SCAVENGE THE ACID THAT IS FORMED AND PREVENT CORROSION TO METALS. WATER MAY REACT WITH THE INHIBITOR AND ALLOW THE NATURAL DECOMPOSITION TO OCCUR.

#### INCOMPATIBILITIES:

METHYL CHLOROFORM (1,1,1-TRICHLOROETHANE):
ACETONE: EXOTHERMIC REACTION.
ALKALI (STRONG): POSSIBLE VIOLENT REACTION.
ALUMINUM AND ALLOYS: HAY DECOMPOSE VIOLENTLY.
BARIUM: FIRE AND EXPLOSION HAZARD.
HAGNESIUM: VIOLENT DECOMPOSITION WITH EVOLUTION OF HYDROGEN CHLORIDE.
METALS (POWDERED): FIRE AND EXPLOSION HAZARD.
NITROGEN TETROXIDE: FORMS EXPLOSIVE MIXTURE.

OXIDIZERS (STRONG): POSSIBLE VIOLENT REACTION.

OXYGEN (GAS): POSSIBLE EXPLOSION WHEN HEATED @ 100 C.

OXYGEN (LIQUID): POSSIBLE VIOLENT EXPLOSION. , POTASH: FORMS FLAMMABLE OR EXPLOSIVE PRODUCT.

POTASSIUM AND ALLOYS: FORMS SHOCK-SENSITIVE MIXTURE.

POTASSIUM HYDROXIDE: FORMATION OF SPONTANEOUSLY FLAMMABLE PRODUCT.

RUBBER, PLASTICS, COATINGS: MAY BE ATTACKED.

SODIUM AND ALLOYS: FIRE AND EXPLOSION HAZARD.

SODIUM HYDROXIDE: FORMS SPONTANEOUSLY FLAMMABLE PRODUCT.

SODIUM-POTASSIUM ALLOY: POSSIBLE EXPLOSION.

TIN AND ALLOYS: INCOMPATIBLE.

ZINC AND ALLOYS: INCOMPATIBLE.

#### **CECOMPOSITION:**

THERMAL DECOMPOSITION PRODUCTS MAY INCLUDE TOXIC AND CORROSIVE FUMES OF CHLORIDES, TOXIC FUMES OF PHOSGENE AND CHLOROACETYLENES, AND OXIDES OF CARBON.

#### POLYMERIZATION:

HAZARDOUS POLYMERIZATION HAS NOT BEEN REPORTED TO OCCUR UNDER NORMAL TEMPERATURES AND PRESSURES.

#### · STORAGE-DISPOSAL

OBSERVE ALL FEDERAL, STATE AND LOCAL REGULATIONS WHEN STORING OR DISPOSING OF THIS SUBSTANCE. FOR ASSISTANCE, CONTACT THE DISTRICT DIRECTOR OF THE ENVIRONMENTAL PROTECTION AGENCY.

#### \*\*STORAGE\*\*

STORE IN A COOL, DRY, WELL-VENTILATED LOCATION, AWAY FROM ANY AREA WHERE THE FIRE HAZARD MAY BE ACUTE (NFPA 49, HAZARDOUS CHEMICALS DATA, 1975).

STORE AWAY FROM INCOMPATIBLE SUBSTANCES.

#### \*\*DISPOSAL\*\*

DISPOSAL MUST BE IN ACCORDANCE WITH STANDARDS APPLICABLE TO GENERATORS OF HAZARDOUS WASTE, 40CFR 262. EPA HAZARDOUS WASTE NUMBER U226.

#### CONDITIONS TO AVOID

MAY BURN BUT DOES NOT IGNITE READILY. CONTAINER MAY EXPLODE IN HEAT OF FIRE.

#### SPILLS AND LEAKS

#### SOIL-RELEASE

DIG A HOLDING AREA SUCH AS A PIT, POND OR LAGGON TO CONTAIN SPILL AND DIKE-SURFACE FLOW USING BARRIER OF SOIL, SANDBAGS, FOAMED POLYURETHANE OR FOAMED CONCRETE. ABSORB LIQUID MASS WITH FLY ASH OR CEMENT POWDER.

#### WATER-SPILL:

LIMIT SPILL MOTION AND DISPERSION WITH NATURAL BARRIERS OR OIL SPILL CONTROL BOOMS.

TRAP SPILLED MATERIAL AT BOTTOM IN DEEP WATER POCKETS, EXCAVATED HOLDING AREAS OR WITHIN SAND BAG BARRIERS.

USE SUCTION HOSES TO REMOVE TRAPPED SPILL MATERIAL.

THE CALIFORNIA SAFE DRINKING WATER AND TOXIC ENFORCEMENT ACT OF 1986 (PROPOSITION 65) PROHIBITS CONTAMINATING ANY KNOWN SOURCE OF CRINKING WATER WITH SUBSTANCES KNOWN TO CAUSE CANCER AND/OR REPRODUCTIVE TOXICITY.

#### OCCUPATIONAL-SPILL:

SHUT OFF IGNITION SOURCES. STOP LEAK IF YOU CAN DO IT NITHOUT RISK. FOR SMALL LIQUID SPILLS, TAKE UP WITH SAND, EARTH OR OTHER ABSORBENT MATERIAL. FOR LARGER SPILLS, DIKE FAR AHEAD OF SPILL FOR LATER DISPOSAL. NO SMOKING, FLAMES OR FLARES IN HAZARD AREA: KEEP UNNECESSARY PEOPLE AWAY.

### REPORTABLE QUANTITY (RD): 1000 POUNDS

THE SUPERFUND AMENDMENTS AND REAUTHORIZATION ACT (SARA) SECTION 304 REDUIRES THAT A RELEASE EQUAL TO OR GREATER THAN THE REPORTABLE QUANTITY FOR THIS SUBSTANCE BE IMMEDIATELY REPORTED TO THE LOCAL EMERGENCY PLANNING COMMITTEE AND THE STATE EMERGENCY RESPONSE COMMISSION (40 CFR 393.40). IF THE RELEASE OF THIS SUBSTANCE IS REPORTABLE UNDER CERCLA SECTION 103, THE NATIONAL RESPONSE CENTER MUST BE NOTIFIED IMMEDIATELY AT (800) 424-8802 OR (202) 426-2673 IN THE METROPOLITAN WASHINGTON, D.C. AREA (40 CFR 302.6).

#### PROTECTIVE EQUIPMENT SECTION

#### VENTILATION:

PROVIDE LOCAL EXHAUST OR PROCESS ENCLOSURE VENTILATION TO MEET PUBLISHED EXPOSURE LIMITS.

#### RESPIRATOR:

THE FOLLOWING RESPIRATORS AND MAXIMUM USE CONCENTRATIONS ARE RECOMMENDATIONS BY THE U.S. DEPARTMENT OF HEALTH AND HUMAN SERVICES, MIOSH POCKET GUIDE TO CHEMICAL HAZARDS OR MIOSH CRITERIA DOCUMENTS; OR DEPARTMENT OF LABOR, 29CFR1910 SUBPART Z.

THE SPECIFIC RESPIRATOR SELECTED MUST BE BASED ON CONTAMINATION LEVELS FOUND IN THE WORK PLACE AND BE JOINTLY APPROVED BY THE MATICNAL INSTITUTE OF

OCCUPATIONAL SAFETY AND HEALTH AND THE MINE SAFETY AND HEALTH ADMINISTRATION.

METHYL CHLOROFORM (1.1.1-TRICHLOROETHANE):

- 1000 PPM- ANY SUPPLIED-AIR RESPIRATOR WITH FULL FACEPIECE.

  ANY SELF-CONTAINED BREATHING APPARATUS WITH FULL FACEPIECE.
- ESCAPE- ANY AIR-PURIFYING FULL FACEPIECE RESPIRATOR (GAS MASK) WITH A CHIN-STYLE OR FRONT OR BACK-MOUNTED ORGANIC VAPOR CANISTER.

  ANY APPROPRIATE ESCAPE-TYPE SELF-CONTAINED BREATHING APPARATUS.
- FOR FIREFIGHTING AND OTHER IMMEDIATELY DANGEROUS TO LIFE OR HEALTH CONDITIONS:
  - SELF-CONTAINED BREATHING APPARATUS WITH FULL FACEPIECE OPERATED IN PRESSURE DEMAND OR OTHER POSITIVE PRESSURE MODE.
  - SUPPLIED-AIR RESPIRATOR WITH FULL FACEPIECE AND OPERATED IN PRESSURE-DEMAND OR OTHER POSITIVE PRESSURE MODE IN COMBINATION WITH AN AUXILIARY SELF-CONTAINED BREATHING APPARATUS OPERATED IN PRESSURE-DEMAND OR OTHER POSITIVE PRESSURE MODE.

#### CLOTHING:

EMPLOMEE MUST MEAR APPROPRIATE PROTECTIVE (IMPERVIOUS) CLOTHING AND EQUIPMENT TO PREVENT ANY POSSIBILITY OF SKIN CONTACT WITH THIS SUBSTANCE.

#### GLOVES:

EMPLOYEE MUST WEAR APPROPRIATE PROTECTIVE GLOVES TO PREVENT CONTACT WITH THIS SUBSTANCE.

#### EYE PROTECTION:

EMPLOYEE MUST WEAR SPLASH-PROOF OR DUST-RESISTANT SAFETY GOGGLES AND A FACESHIELD TO PREVENT CONTACT NITH THIS SUBSTANCE. CONTACT LENSES SHOULD NOT BE WORN.

#### EMERGENCY WASH FACILITIES:

WHERE THERE IS ANY POSSIBILITY THAT AN EMPLOYEE'S EYES AND/OR SKIN MAY BE EXPOSED TO THIS SUBSTANCE, THE EMPLOYER SHOULD PROVIDE AN EYE WASH FOUNTAIN AND QUICK DRENCH SHOWER WITHIN THE IMMEDIATE WORK AREA FOR EMERGENCY USE.

AUTHORIZED BY- OCCUPATIONAL HEALTH SERVICES, INC.

CREATION DATE: 10/25/84

REVISION DATE: 04/12/89

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1.1.1-TRICHLOROETHANE

! ------

THE ENCLOSES MATERIAL SAFETY DATA SHEETS (MSDS'S) ARE SUPPLIES FOR USE BY LABORATORIES RECEIVING U.S. EPA GARN PROJECT ANALYTICAL STANDARDS. SOME OF THESE MSDS'S ARE COPPRIGHTED BY OCCUPATIONAL MEALTH SERVICES, INC. (ONS). USERS OF THE ONS MSDS'S NAY NOT SELL, COPY OR OTHERWISE DISTRIBUTE ANY OF THE MATERIALS, BATA OR SPROMATION STIMES IN PART OR WOLLE OUISIDE OF THESE INTERNAL USER CONSTITUTEMEY.

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FOR A PARTICULAR PURPOSE OR HERCHARTABILITY, NOR ARE SUCH REPRESENTATIONS ON MARRANTIES INPLIES WITH RESPECT !
TO THE DATA FURNISHED. ONS ASSUMES NO RESPONSIBILITY, WITH RESPECT TO CUSTOMER'S, ITS EMPLOYEES', CLIENTS', OR CUSTOMERS' USE TRESEST. ONS SHALL NOT SE LIVELE FOR ANY SPECIAL, CONSEQUENTIAL OR EXEMPLARY DAPAGES
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> Chemtrec # (800) 424-9300 National Response Center # (800) 424-8802



T4940 -01

Trichloroethylene

Issued: 10/15/85

Effective: 10/14/85

Page: 1

SECTION I - PRODUCT IDENTIFICATION

Product Name:

Trichloroethylene

Formula:

C'HC13

Formula Wt: CAS No . :

131.40 00079-01-6

NIOSH/RTECS No .:

KX4550000 Trichloroethene; Ethinyl Trichloride; Acetylene Trichloride;

Common Synonyms:

Product Codes:

5376,9458,9454,9455,9464,9473

PRECAUTIONARY LABELLING 

HEALTH

FLAMMABILITY

REACTIVITY SLICHT



Laboratory Protective Equipment









Precautionary Label Statements

WARNING!

HARMFUL IF SWALLOWED OR INHALED

CAUSES IRRITATION

NOTE: THIS MATERIAL OR ITS VAPORS IN CONTACT WITH FLAMES OR HOT GLOWING

SURFACES MAY FORM CORROSIVE ACID FUMES. NOTE: Reported as causing cancer in laboratory animals.

Exercise due care.

Avoid contact with eyes, skin, clothing.

Avoid breathing vapor. Keep in tightly closed container. Use with adequate

ventilation. Wash thoroughly after handling.

SECTION II - HAZARDOUS COMPONENTS

Component

<u>%</u>

CAS No .

Trichloroethylene

90-100

79-01-6



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Trichloroethylene

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SECTION III - PHYSICAL DATA

Boiling Point:

86°C ( 187°F)

Vapor Pressure(mmHq):

Melting Point:

-73°C ( -99°F)

Vapor Density(air=1): 4.53

Specific Gravity:

Evaporation Rate: (Butyl Acetate=1)

 $(H_2O=1)$ 

Negligible (less than 0.1 %) % Volatiles by Volume: 100

Solubility(H<sub>2</sub>O):

Appearance & Odor: Liquid with chloroform odor.

SECTION IU - FIRE AND EXPLOSION HAZARD DATA

Flash Point:

NFPA 704M Rating: 1-1-0

Flammable Limits: Upper - 10.5 %

Lower - 8 %

Fire Extinguishing Media

Use extinguishing media appropriate for surrounding fire.

L \_\_ial Fire-Fighting Procedures

Firefighters should wear proper protective equipment and self-contained (positive pressure if available) breathing apparatus with full facepiece. Move exposed containers from fire area if it can be done without risk. Use water to keep fire-exposed containers cool.

Unusual Fire & Explosion Hazards

Closed containers exposed to heat may explode.

Toxic Gases Produced

hydrogen chloride

SECTION U - HEALTH HAZARD DATA

Some experiments with test animals indicated that this substance may be anticipated to be a carcinogen.

Threshold Limit Value (TLV/TWA): 270 mg/m<sup>3</sup> (50

ppm)

Short-Term Exposure Limit (STEL): 1080 mg/m<sup>3</sup> (200

LD<sub>sn</sub> (oral-rat)(mg/kg)

4920

LD<sub>50</sub> (ipr-mouse)(mg/kg)

3000

cts of Overexposure

Inhalation of vapors may cause nausea, vomiting, headache, or loss of



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SECTION U - HEALTH HAZARD DATA (Continued)

Ingestion may cause nausea, vomiting and loss of consciousness.

Emergency and First Aid Procedures If swallowed, if conscious, immediately induce vomiting.

If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen.

In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Flush skin with water.

SECTION UI - REACTIVITY DATA

Stability: Stable Hazardous Polymerization: Will not occur

Conditions to Avoid: heat, light, sources of ignition, flame

Incompatibles: chemically active metals, strong bases

Decomposition Products: hydrogen chloride

SECTION UII - SPILL AND DISPOSAL PROCEDURES

Steps to be taken in the event of a spill or discharge

Wear self-contained breathing apparatus and full protective clothing. Stop leak if you can do so without risk. Use water spray to reduce vapors. Take up with sand or other non-combustible absorbent material and place into container for later disposal. Flush spill area with water.

Disposal Procedure

Dispose in accordance with all applicable federal, state, and local environmental regulations.

EPA Hazardous Waste Number: U228 (Toxic Waste)

SECTION UIII - INDUSTRIAL PROTECTIVE EQUIPMENT

Ventilation: Use general or local exhaust ventilation to meet

TLV requirements.

Respiratory Protection: Respiratory protection required if airborne

concentration exceeds TLU. At concentrations up to 1000 ppm, a chemical cartridge respirator with organic vapor cartridge is recommended. Above this level, a self-contained breathing apparatus

is recommended.

Eye/Skin Protection: 🕟 Safety goggles and face shield, uniform,

protective suit, neoprene gloves are recommended.



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SECTION IX - STORAGE AND HANDLING PRECAUTIONS

SAF-T-DATA TM Storage Color Code: Blue

Special Precautions

Keep container tightly closed. Store in secure poison area.

SECTION X - TRANSPORTATION DATA AND ADDITIONAL INFORMATION

DOMESTIC (D.O.T.)

Proper Shipping Name

Trichloroethylene (air only)

Hazard Class

ORM-A

**UN/NA** 

**UN1710** 

Labels

NONE

Reportable Quantity

1000 LBS.

INTERNATIONAL (I.M.O.)

Proper Shipping Name

Trichloroethylene

Hazard Class

'n

UN1710

HARMFUL - STOW AWAY FROM FOOD STUFFS

N/A = Not Applicable or Not Available

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M4420 -01

Methylene Chloride

Page: 1

Effective: 10/14/85

Issued: 10/15/85

SECTION I - PRODUCT IDENTIFICATION

Product Name:

Methylene Chloride

Formula:

CH<sub>2</sub>Cl<sub>2</sub>

Formula Wt:

84.93

CAS No . :

00075-09-2 NIOSH/RTECS No : PA8050000

Common Synonyms: Dichloromethane; Methylene Dichloride; Methane Dichloride

Product Codes:

9324,0480,9315,9329,9264,5531,9330

PRECAUTIONARY LABELLING

BAKER SAF-T-DATA TM Sustem

FLAMMABILITY





SLIGHT Laboratory Protective Equipment









Precautionary Label Statements

POISON! DANGER!

HARMFUL IF INHALED - CAUSES IRRITATION

Reported as causing cancer in laboratory animals. Exercise due care. Avoid contact with eyes, skin, clothing.

Keep in tightly closed container. Wash thoroughly after handling.

SECTION II - HAZARDOUS COMPONENTS

Component

<u>&</u>

CAS No .

Methylene Chloride

90-100

75-09-2

SECTION III - PHYSICAL DATA

Boiling Point:

40°C ( 104°F)

Vapor Pressure(mmHq): 350

Melting Point: -95°C (-139°F)

Vapor Density(air=1): 2.9



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M4420 - 01

Methylene Chloride

Page: 2

Effective: 10/14/85 

Issued: 10/15/85

SECTION III - PHYSICAL DATA (Continued)

Specific Gravity: 1.32

 $(H_{2}0=1)$ 

Evaporation Rate:

27.5

(Butyl Acetate=1)

Solubility(H<sub>2</sub>O):

Moderate (1 to 10 %)

% Volatiles by Volume: 100

Appearance & Odor: Colorless, volatile liquid with penetrating, ether-like odor.

SECTION IU - FIRE AND EXPLOSION HAZARD DATA

Flash Point:

NFPA 704M Rating: 2-1-0

Flammable Limits: Upper - 19 %

Lower -

12 %

Fire Extinguishing Media

Use extinguishing media appropriate for surrounding fire.

Special Fire-Fighting Procedures

Firefighters should wear proper protective equipment and self-contained breathing apparatus with full facepiece operated in positive pressure mode.

Toxic Gases Produced

hydrogen chloride, phosgene

SECTION U - HEALTH HAZARD DATA

Some experiments with test animals indicated that this substance may be anticipated to be a carcinogen.

Threshold Limit Value (TLV/TWA):

 $350 \text{ mg/m}^3 (100)$ 

Short-Term Exposure Limit (STEL): 1740 mg/m<sup>3</sup> (500

Toxicity:

LD<sub>50</sub> (oral-rat)(mg/kg)

2524

LD<sub>50</sub> (ipr-mouse)(mg/kg)

1500

LC<sub>50</sub> (inhal-rat-)(g/m<sup>3</sup>)

88

Effects of Overexposure

Inhalation of vapors may cause nausea, vomiting, lightheadedness or

Liquid may be irritating to skin, eyes, and mucous membranes.

Emergency and First Aid Procedures

If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen.



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National Response Center # (800) 424-8802

| M4420 -01<br>Effective:                   | 10/14/85  | Methylene Chloride   | Page: :<br>Issued: 10/15/8                                 |
|---|---|--|--|
|   | SECTIO  | N V - HEALTH HAZARD DATA (Continued)   |  |
| least                                     | 15 minutes.   | immediately flush eyes with plenty of Flush skin with water.   | •  |
|   |   | SECTION VI - REACTIVITY DATA   | •  |
| Stability:                                |   | Hazardous Polymerization:  |  |
| Conditions                                | to Avoid:   | heat   | •  |
|   | ion Products:   | hydrogen chloride  | =======================================                    |
|   | SECTIO  | N VII - SPILL AND DISPOSAL PROCEDURE   |  |
| Steps to be<br>Wear s<br>Stop 1<br>Take o | e taken in the<br>self-contained<br>seak if you ca<br>up with sand o<br>container for | event of a spill or discharge<br>breathing apparatus and full protect<br>n do so without risk. Use water spra<br>r other non-combustible absorbent mad<br>later disposal. Flush spill area wit | tive clothing.<br>ay to reduce vapors.<br>terial and place |
| Dispos                                    |   | ce with all applicable federal, state ations.  | e, and local   |
|   | us Waste Numb   | er: U080 (Toxic Waste)   |  |
|   | SECTION   | VIII - INDUSTRIAL PROTECTIVE EQUIPMEN  |  |
| Ventilation                               |   | Use general or local exhaust ventile<br>TLV requirements.  |  |
| Respiratory                               | Protection:   | Respiratory protection required if a concentration exceeds TLV. At conceabove 100 ppm, a self-contained breapparatus is advised.   | entrations   |
| Eye/Skin Pr                               | otection:   | Safety goggles and face shield, unit protective suit, polyvinyl alcohol grecommended.  |  |
|   | SECTION   | IX - STORAGE AND HANDLING PRECAUTION   | 45   |
| SAF-T-DATA                                | M Storage Col   | or Code: Blue  |  |
| C   |   |  |  |

Continued on Page:

Keep container tightly closed. Store in secure poison area.

Keep containers out of sun and away from heat.



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M4420 - 01

Methylene Chloride

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SECTION X - TRANSPORTATION DATA AND ADDITIONAL INFORMATION

DOMESTIC (D.O.T.)

Proper Shipping Name

Dichloromethane (air only)

Hazard Class

ORM-A

UN/NA

UN1593

Labels

NONE

INTERNATIONAL (I.M.O.)

Proper Shipping Name

Dichloromethane

Hazard Class

UN/NA

**UN1593** 

Labels

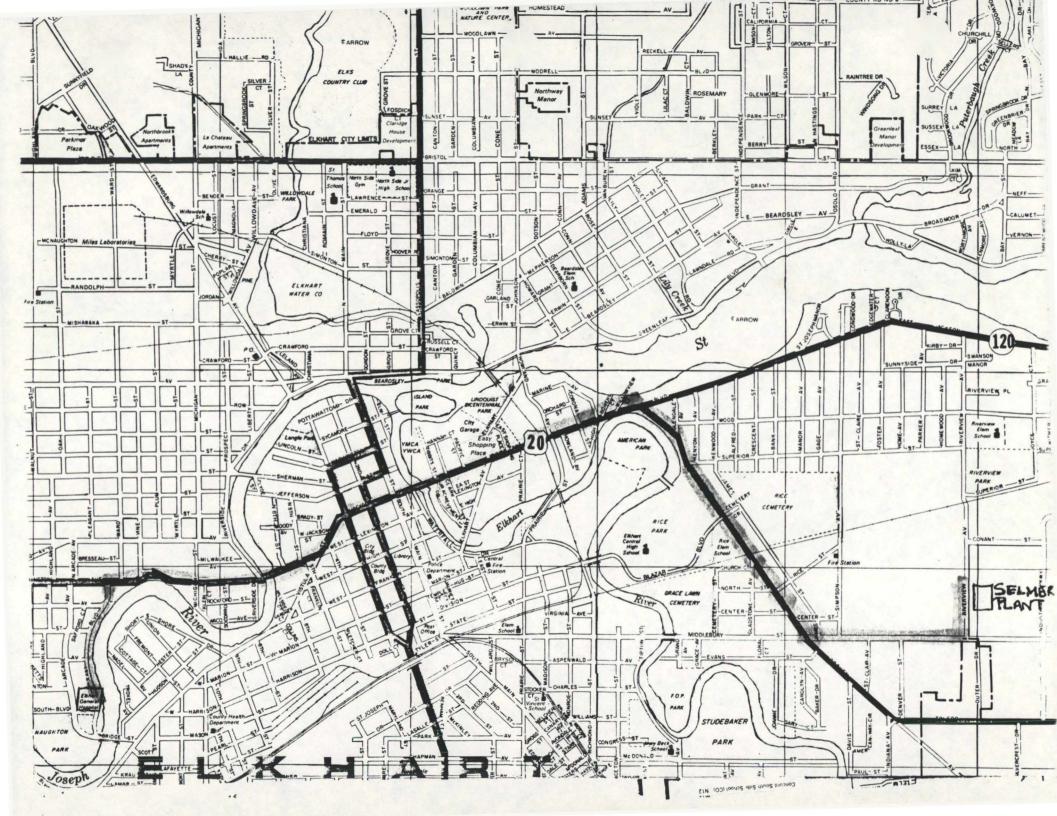
HARMFUL - STOW AWAY FROM FOOD STUFFS

N/A = Not Applicable or Not Available

The information published in this Material Safety Data Sheet has been compiled from our experience and data presented in various technical publications. It is the user's responsibility to determine the suitability of this information for the adoption of necessary safety precautions. We reserve the right to revise rial Safety Data Sheets periodically as new information becomes available.

## Attachment E

Map to the Hospital



Health & Safety Plan

Acknowledgment Form

I have been informed and understand and will abide by the procedures set forth in the Health and Safety Plan and Amendments for the Selmer Plant located in Elkhart, Indiana.

| PRINTED NAME | SIGNATURE | REPRESENTING | DATE |
|--------------|-----------|--------------|------|
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### Appendix C

Standard Operating Procedures for Field Activities

### **WWES SOP**

Field Notes/Records

#### FIELD NOTES/RECORDS

Each week field notes must be copied to the Project Manager, or the Project Engineer/Geologist. Don't invest time in editing or rewriting them; the notes should have been taken throughout the day and, therefore, they should be a sufficiently accurate record in their original form.

The best time to copy the notes would be with the lab copier as you are turning in samples at the lab receiving desk, or, at Eagle Drive when returning equipment.

By providing the copies weekly, the Project Manager will have timely information and can begin to get any of his questions about the notes answered right away.

#### **NOTES MUST INCLUDE:**

- 1) Project Name
- 2) Project Number
- 3) Technician's Name
- 4) Date
- 5) Location of Field Activity (for example, Lansing, corner N. Logan and Sunset)
- 6) Materials and Equipment list, including the quantities on-site at the beginning of each day
- 7) Weather <u>limited</u> information (sunny, overcast, humid, precipitation, wind, approximate temperature)
- 8) Methods, <u>Brief</u> "according to work plan". If deviation from plan, you must note what was done differently, why, and the results. You must note whether the deviation was called for by you, or a departure by the contractor. If by the contractor, whether you have approved or disapproved it.
- Problems Encountered how they were dealt with. Problems include equipment malfunction, delays, unsafe working conditions or procedures, departure from the Health & Safety Plan, attitudes/comments of workers/visitors, weather adversely affecting the work, and inspected work found to be unsatisfactory. Include a sketch of an unusual procedure, if helpful.
- 10) Visitors who and when, their comments
- 11) Location Descriptions wells, borings, and sampling stations. Distance tie-in to two fixed site facilities.

12) Summary of Work Accomplished - For example, locations completed (such as borings B-1, B-9; well 3-A), soil volume removed, depth of drilling in progress.

Concerning the numbered items of information above:

- Record Items 1 through 7 right away. Item 6 is optional, depending upon the manager and project needs.
- Record Items 7 through 11 during the day, as any changing weather and site events call for. Record Item 11 only if locations are not documented elsewhere.
- Attend to Item 12 at the end of the working day.

Notes must reference any forms used for documenting calculations, location descriptions, depth measurements, and time and materials. For example:

Sampling Calculations--see attached WSFR form

Well/Boring Elevations--see attached survey form

Water Level Data--see attached WL & FR forms

Time and Materials--see attached DT & MR form

Attach the original filled-out forms with copies of the notes.

Soils Classification

#### **SOILS CLASSIFICATION**

There are several different soil classification systems. In order to maintain a level of consistency on our logs, WWES has adopted, in general, the Unified Soils Classification System (USCS). By using this system we are able to maintain a consistent soil and well log database with a widely accepted soils classification system so that the information in the database can be readily accessed and interpreted by many people. Use of the generalized symbols of the USCS accelerates soil correlations and the production of geologic cross sections through integrated computer software programs.

This system was originally proposed in 1942 for use in the air field construction works undertaken by the Army Corps of Engineers, during World War II. It was developed in 1948 and modified in 1952. Its origin is based on engineering design concepts.

Most soil descriptions are written in the field based on visual and manual observations, but if a more accurate description is required, than the only laboratory analyses that are required to definitively classify the soil sample according the USCS are a sieve analyses and the Atterberg limits.

There are four major soil divisions in the USCS:

- 1. Coarse grained,
- 2. Fine grained,
- 3. Organic soils, and
- 4. Peat

Soils too large to pass through a 75 mm sieve (about 3 inches) are considered "oversized" material according to this system. These "oversized" materials are the boulders and the cobbles.

Each soil description should follow this general order:

- 1. Texture
  - a. basic
  - b. modifying
- 2. Consistency
- 3. Color
- 4. Moisture content

After the moisture, or water, content of the soil has been described, further observations-such as odor, presence of roots or debris, or any other notable observations--are recorded.

Each of these items is described in more detail below.

#### 1. Texture

#### a. Basic Texture

| USCS<br><u>Division</u> | Basic Texture or Description to Use | Particle Diameter Size  | Common<br>Comparison                                   |
|-------------------------|-------------------------------------|---|--|
| Oversized<br>Soils      | Boulder<br>Cobble                   | larger than 12 inches<br>3 to 12 inches                       |  |
| Coarse grained Soils    | Coarse gravel<br>Fine gravel        | 3/4 to 3 inches<br>4.75 mm to 3/4 inch                        | pea to large marble                                    |
|                         | Coarse sand<br>Medium sand          | 2 to 4.75 mm<br>.425 mm to 2 mm                               | pepper to pea size<br>pencil lead to pepper            |
|                         | Fine sand                           | .075 to .425 mm   | table sugar  |
| Fine grained Soils      | Silt<br>Clay                        | <.075 mm<br><.075 mm  | powdered sugar<br>individual grains are<br>not visible |
| Highly Organic<br>Soils | Peat                                | organic, fibrous or<br>amorphous textured<br>vegetable tissue |  |
|                         | Organics                            | define as muck, coal, etc.                                    |  |

Clay is distinguished from silt by plasticity. Silt is non plastic or very slightly plastic and exhibits little or no strength when air dry (plasticity index <4). Clay can be made to exhibit plasticity (putty-like properties) within a range of water content, and it exhibits considerable strength when air dry (plasticity index >4).

There are a couple of tests to use in the field to distinguish clay from silt. In a moist sample, clay will ribbon down to a thickness of approximately 1/32 of an inch and can be molded into any shape.

As the silt or sand content in a clay increases, so does the ribbon thickness and molding the sample becomes more difficult. A moist silt sample will ribbon to only 1/4 inch or greater in thickness and is more difficult to mold.

In wet soils clay, when rubbed into the palm of a hand, is very difficult to rub off. Clay will hold the water content when shaken. When a wet silt drys in the hand, it can be rubbed off readily and a wet silt sample will puddle readily when shaken. See Tables 4 and 5 for field guidance to distinguish silts from clays.

#### b. Modifying Texture

Estimates of the modifying texture are given using the following adjectives:

For sands and finer grained particles:

| Descriptive |                             |
|-------------|-----------------------------|
| Word        | <b>Estimated Percentage</b> |
| Trace       | less than 10%               |
| Little      | 10 to 30%                   |
| Some        | 30 to 45%                   |
| And         | 45 to 50%                   |

For particles coarser than gravels (i.e., cobbles and boulders), the adjective "occasional" may be used to describe their percentage. An estimate of the maximum grain size should also be stated in the description for coarse gravel and larger grain sizes.

#### 2. Consistency

The consistency of sands or gravels is described adequately by the blow counts required to drive the split-spoon sampler. Therefore, if the blow counts are recorded, the description of consistency may be omitted. For fine grained soils (i.e. clays), the description of consistency in addition to the recorded blow counts is more informative. For clay, use the following descriptions to define the consistency.

| Description | <u>Criteria</u>  |
|-------------|--|
| Very hard   | Thumbnail will not dent  |
| Hard        | Thumb will not indent soil but readily indented with thumbnail |
| Firm        | Thumb will indent soil about 1/4 inch                          |
| Soft        | Thumb will penetrate soil about 1 inch                         |
| Very soft   | Thumb will penetrate soil more than 1 inch                     |

The consistency of clays can be defined in the laboratory by the results of the unconfirmed compression test.

#### **Plasticity**

Refer to Table 4 and especially the section on *toughness* for a practical approach to estimation of plasticity (consistency near plastic limit) in the field; and based on that, to assign "H" or "L" to cohesive organic samples.

#### 3. Color

Color is useful in distinguishing soils similar in geologic origin. Record the color of the soil as you see it. The color may be modified by an adjective (e.g. light brown) If there are two major but distinct colors in the soil, describe the color as mottled or variegated (e.g., gray mottled brown).

#### 4. Moisture Content

Describe the water content of every soil type encountered. Three main adjectives to use are:

| Water Content | Sample Characteristics            |
|---------------|-----------------------------------|
| Dry           | Powdery or hard                   |
| Moist         | Plastic or containing some liquid |
| Wet           | Saturated or puddles when shaken  |

Again, modifiers can be added to describe varying degrees of moisture content.

Once the soil boring is complete and all the soil types have been described, assign the appropriate USCS symbol to each soil description on the soil log so that similar soils can be clearly grouped together to represent the major soil layers present at the site (Tables 1, 2 and 3).

#### **Examples:**

| Soil Description  | USCS Symbol    |
|---|----------------|
| SAND, fine to medium, some silt, trace clay, light brown, wet                                       | SM             |
| CLAY, some silt, trace medium to fine sand, soft, brown mottled a moist (trace rootlets)            | gray,<br>ML/CL |
| SILT, trace fine sand, yellow-brown, dry  | ML             |
| SAND AND GRAVEL, brown, dry   | SW/GW          |
| CLAY, little silt, trace fine to medium sand, occasional cobble (6" very hard, gray, slightly moist | CL             |
| PEAT, brown, very soft, wet   | PT             |
| SILT, some clay, little organics, very soft, moist (decaying odor)                                  | OL/OH          |

A borderline symbol is two symbols separated by a slash, for example OL/OH. A borderline symbol is used to indicate a soil that has been identified as having properties that do not distinctly place the soil into a specific group.

Each log should be accurate and detailed. The frequency of soil sampling should be reflected in the detail of the well/boring log. The log for a borehole sampled continuously will be much more detailed in both soil descriptions and depths than a log for the borehole in which no soil samples were collected.

Well/boring log forms must be completed for every well or boring installed, regardless of depth or method used, even if it has not been specifically requested.

#### References:

1991 Annual Book of ASTM Standards, Volume 04.08 D 2488-90 "Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)

An Introduction to Geotechnical Engineering Robert D. Holtz, William D Kovacs, Prentice-Hall Civil Engineering Mechanics Series

AGI Data Sheets, 3rd Edition Compiled by J.T. Dutro, Jr., R.V. Dietrich, R. M. Foose, American Geological Institute 1989, Data sheets 29.2 and 38.1

Earth Manual, A Water Resources Technical Publication, 2nd ed., U.S. Department of the Interior, United States Government Printing Office Washington 1974. Reprinted in 1980.

A-17

**VERSION 2: 1992** 

#### TABLE 4

#### FIELD IDENTIFICATION PROCEDURES FOR FINE GRAINED SOILS1

These procedures may be performed on the minus No. 40 sieve size particles, approximately 1/64 inch for field classification purposes, screening is not intended, simply remove by hand the course particles that interfere with the tests.

DILATANCY: (Reaction to shaking)

Take a small amount of moist soil. Add enough water, if necessary, to make the soil soft but not sticky. Place the soil in the open palm of one hand and shake horizontally, striking vigorously against the other hand several times. A positive reaction consists of the appearance of water on the surface of the soil which changes to a livery consistency and becomes glossy. When the sample is squeezed between the fingers, the water and gloss disappear from the surface, the sample stiffens, and finally it cracks or crumbles.

The rapidity of the appearance of water during shaking and of its disappearance during squeezing assist in identifying the character of the fines in a soil.

Very fine clean sands give the quickest and the most distinct reaction whereas a plastic clay has no reaction. Inorganic silts show a moderately quick reaction.

#### DRY STRENGTH (Crushing characteristics)

Mold a small sample of soil to the consistency of putty, adding water if necessary. Allow the soil to dry completely by sun or air drying, and then test its strength by breaking and crumbling between the fingers. This strength is a measure of the character and quantity of the colloidal fraction contained in the soil. The dry strength increases with increasing plasticity.

High dry strength is characteristic for clays of the CH group. A typical inorganic silt possesses only very slight dry strength. Silty fine sands and silts have about the same slight dry strength, but can be distinguished by the feel when powdering the dried specimen. Fine sand feels gritty whereas a typical silt has the smooth feel of flour.

#### TOUGHNESS (Consistency near plastic limit)

Mold a small sample to the consistency of putty adding water if necessary. If the sample becomes too sticky, spread the sample out in a thin layer and allow some of the soil moisture to evaporate. Roll the specimen out by hand on a smooth surface or between

Earth Manual, A Water Resources Technical Publication, Second Edition, U. S. Department of the Interior, United States Government Printing Office Washington 1974, Reprinted in 1980

the palms into a thread about one-eighth inch in diameter. The thread is then folded and rerolled repeatedly. During this manipulation, the moisture content is gradually reduced and the specimen stiffens, finally loses its plasticity, and crumbles when the plastic limit is reached.

After the thread crumbles, the pieces are lumped together and a slight kneading action continued until the lump crumbles.

The tougher the thread near the plastic limit and the stiffer the lump when it finally crumbles, the more potent is the colloidal clay fraction in the soil and the higher is the plasticity. Weakness of the thread at the plastic limit indicates either inorganic clay of low plasticity, or materials such as Kaolin-type clay and organic clays.

Highly organic clays have a very weak and spongy feel at the plastic limit.

Soil Boring Drilling Using Hollow-Stem Augers

#### SOIL BORING DRILLING USING HOLLOW-STEM AUGERS

#### **Introduction:**

When the primary objective of the drilling is to obtain soil samples from discreet depths, the hollow stem augering (HSA) technique of drilling is one of the most effective. The soil is penetrated with five-foot-long, continuous helical flight augers which are driven by a rotary drive head mounted on a hydraulic feed system which pushes the drill stem down or pulls it up. Cuttings are mechanically removed from the borehole by the flights on the HSA's.

#### Goal:

To drill a soil boring from which the depths and descriptions of the soils encountered can be accurately logged and to obtain samples of the soils from accurate soil depth intervals.

#### Task-Specific Equipment Needed:

1. All drilling equipment and labor are supplied by the subcontracted drilling company.

#### **Procedure:**

- 1. Access the drill rig and all necessary equipment to the proposed borehole location.
- 2. Advance the HSA's to the top of the proposed soil sampling depth.
- 3. With the augers in place and at rest, remove the center plug from the inside of the augers.
- 4. Attach a decontaminated split-spoon sampling or other device to the drilling rods.
- 5. Lower the device inside the HSA to the bottom of the borehole.
- 6. Drive the device into the soil as described, for example, in the WWES' Standard Operating Procedure (SOP) for "Split-spoon Sampling".
- 7. Recover the device.
- 8. Replace the plug inside the HSA and continue drilling to the next sampling depth.
- 9. When the desired completion depth has been attained, properly backfill the borehole from the bottom up as described in WWES's SOP for "Soil Boring Grouting" and decontaminate the drilling and sampling tools according to the SOP "Decontamination, Downhole Sampling Equipment".

Unless there is a specific need for another size auger, WWES's standard is to drill soil borings with 4.25 - inch inside diameter HSA's. The outside diameter of these augers is 8.25 inches.

Well/Boring Log Guidelines

**VERSION 1: 1990** 

#### WELL/BORING LOG GUIDELINES

For every well or soil boring, a separate well/boring log sheet must be completed using our standardized well/boring log sheet. On the top half of the sheet are a number of headings with accompanying blanks. It is imperative that all applicable information in this top area be filled out completely for each well or boring.

Well/Boring No.: The numbering sequence is generally provided by the project

manager and must be recorded accordingly.

Client: Record the client or project name.

County-Township-

Fraction-Section: This information can be filled out at the office by the project

geologist.

Contractor: Give the name of the contractor. Include their complete address.

The equipment used should be documented. If a drill rig was used, record the make and the model. The name of the drilling crew chief should also appear here. The supervisor would be the

WWES person responsible for overseeing the work in the field.

Drilling Methods: Record the method of drilling that was used. Also, record the

diameters of the drill string. Some examples would be:

- 4 1/4" I.D. HSA (inside diameter hollow stem auger)

- 10" O.D. HSA (outside diameter hollow stem auger)

- 3-1/8" mud rotary

5-1/2" O.D. x 3-1/4" I.D. dual wall reverse air circulation

4" O.D. hand auger

Grouting/Seal: Record the grouting material and the grouting method. If an

additive is used, estimate its weight percentage. Also, record the

bottom and top depths of the grout.

Development: Include the developing method, rate, pumping time, and total

volume evacuated from the well.

Screen: Each item needs to be completed as described on the sheet.

**VERSION 1: 1990** 

Casing:

Record the casing material, diameter, and the bottom and top depths of the casing. A "+" value indicates above-ground well stick-up. An "0" value indicates a flush-mount well. Record the top of well thread distance to grade to the nearest 0.1 foot.

Date:

Record as indicated on the sheet.

Elevation:

Survey elevation data should be recorded here if you have this information. Be sure to include the reference point whether it is U.S.G.S. datum or a relative elevation. Include the location of your reference point. This information may not always be available to you as the drilling takes place, and it may need to be filled in at a later date by others.

Water Level:

In a soil boring, record the first water-saturated level encountered and the elapsed time before measurement. In a well, record the water level using the top of casing as the reference point and note TOC. Also, record the date of the measurement, the time elapsed since development, and the method of measurement.

Location:

Reference your sketch and measurements to features evident on the base map which the project manager has supplied. If no base map has been supplied, reference your measurements to permanent site structures.

Well Sketch:

Somewhere on the log sheet, a sketch of the well construction should be drawn. On this sketch, show the amount of aboveground stick-up, the depths to joints along the well casing, and the depth of the top and bottom of the well screen.

Remarks:

Any added comments that are unique to the boring or well can be recorded here.

On the lower half of the well/boring log sheet is an area for the lithologic description. The soils or rocks that are penetrated during drilling should be recorded as accurately as possible according to the soils classification guidelines. On the left side of the log sheet, a number should be recorded in the thickness and the depth to base columns for every lithologic change recorded in the lithologic descriptions.

The remarks area in this lower portion should be used to clearly identify soil sample depths, blow counts, and any other readings or measurements taken at individual depth intervals (Hnu, OVA).

Split-Spoon Sampling

#### SPLIT-SPOON SAMPLING

Soil borings that are drilled for a geotechnical study (i.e., a study designed to determine the compressive strength of the soil for the purpose of new building construction) are usually sampled at 2.5-foot intervals in the first 10 feet below grade and at 5-foot intervals thereafter to the bottom of the boring.

The depths from which soil samples are collected in an environmental investigation can be very site-specific. Soil samples are often collected from depths most likely to show environmental impact based on an evaluation of the known or suspected contaminants, the characteristics of the soils, and other variables that may affect a particular site.

Collecting a soil sample with a split-spoon or split-barrel sampling device is a common technique used to determine the physical soil characteristics and soil quality. It is described by ASTM Method D-1586 and is summarized below.

#### **JOB DESCRIPTION:**

Obtain soil samples at specified intervals, and collect the soil for laboratory analyses.

#### TASK-SPECIFIC EQUIPMENT NEEDED:

- drilling and sampling devices
- · tape for locating
- · well/boring log sheets

#### **EXPECTATIONS:**

A well/boring log sheet will be accurately completed at each well or boring, according to WWES's standard operating procedure "Well/Boring Log Guidelines", including blow counts, PID or FID response, soil descriptions, and other soil boring details.

The boring will be located by measurements and labeled on a site sketch or base map.

#### **PROCEDURE:**

- 1. Advance the boring to the desired sampling depth.
- 2. Attach the split-spoon sampling device to the bottom end of the drilling rods and gently lower it to the bottom of the borehole.

- 3. A 140-pound hammer free-falling a distance of 30 inches is used to drive the 2-inch O.D. split spoon 18 inches into the undisturbed soil below. Drive the 2-inch O.D. split-spoon sampler into the undisturbed soil ahead of the lead auger.
- 4. Record the number of blows required to drive the sampler for each 6-inch increment. If the soil is particularly hard and the blow counts are in excess of 100 blows per 6 inches, a split spoon may not be capable of obtaining the sample. Stop to keep from damaging the sampling device.
- 5. Bring the split spoon back to the ground surface after it has been driven over the sample interval.
- 6. Open the split spoon.
- 7. Field screen the sample if it is required.
- 8. Take samples that are collected for lab analyses from the mid to lower portions of the split spoon. Immediately place the soil that is most likely to be impacted (based on PID or FID response and visual observations of staining) into the appropriate sample bottles. Collect the samples to be analyzed for volatile organic compounds first. Collect the soil for semi-volatile analyses next, and collect soil for inorganic analyses last.
- 9. Place the samples in appropriate containers, using a clean tool and/or clean gloves.
- 10. Visually inspect the sample and describe it accurately and completely on the well/boring log sheet.

The upper portion of soil in the sampler can be disturbed or not representative of the sample interval targeted. This is because residual soils from within the auger stem may become entrained in the sample. The upper portion should be observed but field judgment should be used as to whether it is really representative of the sample interval. The upper portion should not be collected for lab analyses.

Jar Headspace Measurements In Unsaturated Soil Samples (Using FID or PID)

#### JAR HEADSPACE MEASUREMENTS IN UNSATURATED SOIL SAMPLES

#### (USING FID OR PID)(1)

#### **Introduction:**

This procedure is most commonly used at sites where there is a suspected impact from gasoline constituents. The two instruments most commonly used for this field procedure are a flame ionization detector (FID) and a photoionization detector (PID). The FID response is uniform for most volatile gasoline hydrocarbons while the PID response increases for the BTEX compounds. Therefore, the PID may be more effective when concentrating on the aromatic constituents of gasoline.

Most field devices are sensitive to changing weather, and the response of the PID may become significantly affected by an increase in the humidity.

The FID systems, unlike the PID systems, will respond to methane.

#### Goal:

To obtain a field estimate of the relative concentrations of total volatile organic compounds (VOC's) contained within a soil sample.

#### **Task-Specific Equipment Needed:**

- 1. Flame ionization detector (FID) or a photoionization detector (PID) equipped with a 10.2 eV lamp (HNU brand) or 10.0 ev lamp (Thermo Environmental Instruments OVM/Datalogger).
- 2. Glass sample jars between 9 and 16 ounces in total capacity.
- 3. Aluminum foil.

#### Procedure:

- 1. Calibrate the FID or the PID as indicated in the instrument manual.
- 2. Record the calibration procedure and the calibration results in the field notes. If a dedicated log book accompanies the instrument, record the calibration details in it.
- 3. Collect the soil sample.
- 4. Place the soil sample into the glass sample jar immediately. Fill the sample jar half-full.
- 5. Seal the sample jar by placing a clean piece of aluminum foil over the mouth and threads of the jar.
- 6. Allow the sample to reach approximately 70°F.
- 7. After a 5- to 10-minute headspace development period, vigorously agitate the sample jar for at least 30 seconds.
- 8. Immediately insert the probe of the FID or PID through the aluminum foil seal and into the sample jar.
- 9. Record the maximum meter response as the TOTAL ORGANIC VAPOR HEADSPACE concentration on the Well/Boring log form or the field notes as appropriate.
- 10. Record any significant changes in the weather (and the apparent humidity) that occur throughout the day.

<sup>(1)</sup> John Fitzgerald, Petroleum Contaminants in Soil, Vol. II, pp. 119-135.

### Appendix D

Field and Laboratory Forms

Appendix L

### NERI Chain-of-Custody Record

## NORTHEAST RESEARCH INSTITUTE, INC. Chain of Custody Document

| Tob Number                                      | (Please referance) and shipmen        |              | number | with all cor                          | respondence |
|---|---------------------------------------|--------------|--------|---------------------------------------|-------------|
| .IELD DATA: Facility                            |                                       | <del> </del> |        |                                       | <del></del> |
| Location  |                                       |              |        | <del></del>                           |             |
|   | <u> </u>                              | ·.           |        | · · · · · · · · · · · · · · · · · · · |             |
| Field Manager                                   |                                       |              | 1      | Phone                                 |             |
| CpD LAB DATA:                                   |                                       | GC/MS LAB    | DATA:  |                                       |             |
| Instrument                                      | ·                                     | <del></del>  |        | · · · · · · · · · · · · · · · · · · · |             |
| Operator  |                                       |              |        |                                       |             |
| Phone   |                                       |              | ···    | <del></del>                           |             |
| Sample Nos                                      | · · · · · · · · · · · · · · · · · · · |              |        |                                       | <del></del> |
| SAMPLE DATA:                                    |                                       |              |        |                                       |             |
| Number of Samples                               | 1                                     |              |        |                                       |             |
| Number ReceivedReceived By                      |                                       |              |        |                                       |             |
| SAMPLE TRANSFER DATA:  Relinquished By: Reli 1. | inguished To:                         | <u>Date:</u> | Time:  | Reason:                               |             |
| 2.  |                                       |              | •      |                                       |             |
| 3.  |                                       |              |        | •                                     |             |
| 4.  | -                                     |              |        |                                       |             |
| 5.  |                                       |              |        |                                       | · ,         |
| 6.  |                                       |              |        |                                       |             |

## NERI PETREX® Sample Submittal Form

# Please, return with samples when pickup is complete. PETREX SAMPLE SUBMITTAL FORM

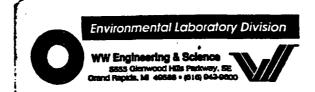
| NE           | RI PROJECT NUMBER:                      |   |           | Γ        | ATE | E:       | (           |       |
|--------------|---|---|-----------|----------|-----|----------|-------------|-------|
|              | RI PROJECT MANAGER:                     | •   | CLIENT    | PROJE    | CT  | MANAGER: |             |       |
| 1)           | TOTAL NUMBER OF TUBES I                 | ENCLOSED:   |           | _        |     |          |             |       |
| 2)           | GREATEST SAMPLE NUMBER:                 |   |           | _        |     |          | <del></del> |       |
| 3)           | MISSING SAMPLE NUMBERS                  | •   |           | · · · ·  |     |          |             | ·<br> |
| _            |   |   |           | ,        |     |          |             |       |
| 4)           | DATE SHIPPED FROM FIELD                 | D:  |           | _        |     | ·        |             |       |
| 5)           | TRAVEL BLANKS:                          |   |           |          |     |          |             |       |
| <u>-</u>     | NOTES:                                  |   |           | <u> </u> |     |          |             |       |
| <del>-</del> |   |   |           |          |     |          |             |       |
|              |   |   |           |          |     | ,        |             |       |
|              | 60                                      | RI-West, ATT<br>5 PARFET STI<br>LAKEWOOD, CC<br>(303) 2 | REET, SUI | TE 10    |     |          |             |       |
|              |   | LAB U   | SE ONLY   |          |     |          |             |       |
| 7)           | REPLICATE SAMPLES:                      | ·   |           |          |     |          |             | ·     |
| _            |   |   |           |          |     |          |             |       |
| 8)           | LAB NOTES:                              |   |           |          |     |          |             |       |
|              | • |   |           |          |     | •        |             |       |
| 9)           | QA/QC BATCH NUMBERS:                    |   |           |          |     |          |             |       |
| DA'          | TE RECEIVED BY LAB/BY: _                |   | V - 1/4   |          |     |          |             |       |
|              | re analyzed/by:                         |   |           |          |     | . •      |             |       |

### WWES Well/Boring Log Sheet

### WW Engineering & Science, Inc. Page: \_\_\_\_\_ of \_\_\_ **Environmental Services Division** Well/Boring No .:\_ Client:\_\_ Project No .: \_ Date: **Well/Boring Log Sheet** Started: \_\_\_ \_ Finished: \_ T Section R County Township Fraction 1/4 1/4 1/4 Location Contractor\_ Address: \_\_\_ Equipment: \_\_\_\_ Crew Chief:\_ WW Supervisor:\_\_\_ Drilling Method(s) Depth Remarks Grouting/Seal Material/Method Depth/To Water Level \_\_\_\_\_ Ft. Below \_ PID/FID (feet) Blow Thick-Depth to Counts Sample Depth 6" 12"18" 24" LITHOLOGIC DESCRIPTION ness Base

Project Name: **WW Engineering & Science** Project No: Log of Well Installation Top of Casing Elevation: Well Designation: Ground Surface Date(s) of Installation:\_\_\_\_\_\_ To:\_\_\_\_\_ Elevation: **Water Level Data** Date Water Level Below TOC Generalized Subsurface Length of Casing Profile **Above Ground Surface** Concrete Cap (Y/N) Development : \_\_\_\_\_ Depth to Top of Grout/Backfill (circle one) Grout Material Survey Reference: Diameter: Total Length: Well Material: Casing Depth to Top of Cap Type: Bentonite Pellets/Slurry (Circle One) Depth to Top of Filter Pack Diameter: Type Length: Well Screen Slot/Type: \_\_\_\_\_ Depth to Bottom Material: of Well Screen Borehole Material: \_\_\_\_\_ Dia. \_\_\_\_ Backfill Materia Protective Height Above Ground: \_\_\_\_\_ Well Casing Total Depth of Borehole Lock Type:\_\_ General Notes:\_\_\_

### WWES Laboratory Sample Tag



| Client:               |          | —    |
|-----------------------|----------|------|
| Project Number: Date: |          |      |
| Preservative:         |          |      |
| Sampled By:           | ·        |      |
| Sample Location:      | <u> </u> | <br> |

WWES Chain-of-Custody Record and Sample Inventory and Master Bottle Packing List

# WW Engineering & Science, Inc. Environmental Laboratory Division 5555 Glenwood Hills Pkwy SE • P.O. Box 874 • Grand Rapids, MI 49512-0874

### **Chain of Custody Record**

COC No.

Νō

44931

|                 |                              |       |              |         |       |              |   |       |        |        |               |        |   |  | _    |                      | ·   |   |                    |               |                  |                       |  |
|-----------------|------------------------------|-------|--------------|---------|-------|--------------|---|-------|--------|--------|---------------|--------|---|--|------|----------------------|---|---|--------------------|---------------|------------------|-----------------------|--|
| WWES Proj.      | Mgr.                         | Proje | ct Name      |         |       |              |   |       |        |        |               |        |   |  |      |                      | No's<br>Correspond to                                       |   | •                  |               | For Lab Use Only |                       |  |
| WWES Proj.      | No.                          | Samj  | pler (Print) |         | 1     | <u>. l</u> . | J |       | Γ.     | Г      | 1             | $\top$ |   |  |      |                      | Bottle Packing<br>List                                      |   |                    |               | Rack/Tray No:    |                       |  |
|                 | Sampler Signature  Date Time |       |              |         |       | _            |   |       |        | ·      | Lab Project # |        |   |  |      |                      |   |   |                    |               |                  |                       |  |
| Date<br>Sampled | Tin<br>Samp                  |       | Matrix*      | Company | 1     | -            |   | •     | Samp   | ple Id | entific       | ation  | 1 |  |      | No. of<br>Containers | Container<br>Type   |   | Analysis Required/ | Comments      | Sample No.       | Filtered<br>Date/Time |  |
|                 | -                            |       |              |         |       |              |   |       |        |        |               |        |   |  |      |                      | 1 2 3 4 5<br>6 7 8 9 10<br>11 12 13 14 15<br>16 17 16 19 20 |   |                    |               |                  |                       |  |
|                 |                              |       |              |         |       |              |   |       |        |        |               |        |   |  |      |                      | 1 2 3 4 5<br>6 7 8 9 10<br>11 12 13 14 15<br>16 17 16 19 20 |   |                    |               |                  |                       |  |
|                 |                              |       |              |         |       |              |   |       |        |        |               |        |   |  |      |                      | 1 2 3 4 5<br>6 7 8 9 10<br>11 12 13 14 15<br>16 17 18 19 20 |   |                    |               |                  |                       |  |
|                 |                              |       |              |         |       |              |   |       |        |        |               |        |   |  |      |                      | 1 2 3 4 5<br>6 7 8 9 10<br>11 12 13 14 15<br>16 17 18 19 20 |   |                    |               |                  |                       |  |
|                 |                              |       |              |         |       |              |   |       |        |        |               |        |   |  |      |                      | 1 2 3 4 5<br>6 7 8 9 10<br>11 12 13 14 15<br>16 17 18 19 20 |   |                    |               |                  | -                     |  |
|                 |                              |       |              |         |       |              |   |       |        |        |               |        |   |  |      |                      | 1 2 3 4 5<br>6 7 8 9 10<br>11 12 13 14 15<br>16 17 18 19 20 |   |                    |               |                  |                       |  |
|                 |                              |       |              |         |       |              |   |       |        |        |               |        |   |  |      |                      | 1 2 3 4 5<br>6 7 8 9 10<br>11 12 13 14 15<br>16 17 18 19 20 |   |                    |               |                  |                       |  |
|                 |                              |       |              |         |       |              |   |       |        |        |               |        | - |  |      |                      | 1 2 3 4 5<br>6 7 8 9 10<br>11 12 13 14 15<br>16 17 18 19 20 |   |                    |               |                  |                       |  |
|                 |                              |       |              |         |       | ,            |   |       |        |        |               | ·      |   |  |      |                      | 1 2 3 4 5<br>6 7 8 9 10<br>11 12 13 14 15<br>16 17 18 19 20 | } |                    |               |                  | ·                     |  |
| Relinquished    | Ву:                          |       |              | Da      | te/Ti | me           |   | Recei | ived B | By:    |               |        |   |  | Rece | ived to I            | ab By:  | • | Date/Time          | Logged in By: |                  | Date/Time             |  |

<sup>\*</sup> Matrix: Water (WTR), Wastewater (WW), Soil (SOIL), Sludge (SLG), Air, Oil, Waste (WASTE) ESL/Charts/Chain of Cuzody

| Bottles will be Shipped / Picked Up  If Shipped: Other Other Ship Bottles to:  Prequency: One Time Quarterly Semi Annual Monthly Annual  Prepare Bottles for:     |             |  |  |
|---|-------------|--|--|
| Bottles Requested on:   |             |  |  |
| Bottles will be Shipped / Picked Up  If Shipped:  |             |  |  |
| If Shipped: Overnight   | Time Due: - |  |  |
| Frequency: One Time Quarterly Others Weekly Semi Annual Annual Annual Outhers Monthly Annual Sep Oct Nov Dec  |             |  |  |
| Frequency: One Time Quarterly Others Weekly Semi Annual Annual Prepare Bottles for:  Months Jan Feb Mar April May June July Aug Sep Oct Nov Dec  Weeks: 1 2 3 4 5 |             |  |  |
| Weekly Semi Annual  Monthly Annual  Prepare Bottles for:  Months Jan Feb Mar April May June  July Aug Sep Oct Nov Dec  Weeks: 1 2 3 4 5                           |             |  |  |
| Months  |             |  |  |
|   |             |  |  |
| Day: DM DT DW DTH DF  |             |  |  |
|   |             |  |  |
| Expiration Date:  |             |  |  |
| COMMENTS  |             |  |  |
|   |             |  |  |
|   |             |  |  |
|   |             |  |  |
|   |             |  |  |
|   |             |  |  |
|   |             |  |  |
| Bottle Prep Use Only  |             |  |  |

Page 1

| Client Name:  |  |  |
|---------------|--|--|
| Olletti Name. |  |  |

For any questions regarding these bottles, contact Ron Hamilton, the Project Chemist for this submittal.

## **Sample Inventory and Master Bottle Packing List**

| Sample                            | Sample Sub-Portions-Preservative and Tagging Codes |     |   |   |   |   |   |     |   |    |    |    |    |    |    |    |    |     |    |     |     |    |    |    |   |
|-----------------------------------|--|-----|---|---|---|---|---|-----|---|----|----|----|----|----|----|----|----|-----|----|-----|-----|----|----|----|---|
|                                   | 1  | 2   | 3 | 4 | 5 | 6 | 7 | 8   | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18  | 19 | 20  | 21  | 22 | 23 | 24 | 2 |
|                                   |  | -   |   |   |   |   |   |     |   |    |    |    |    |    | ,  |    |    |     |    |     |     |    |    |    | - |
|                                   |  |     |   |   |   |   |   |     |   |    |    | -  |    |    |    | 3  |    | 100 |    |     |     |    |    |    | - |
|                                   |  |     |   |   |   |   |   |     |   |    |    |    |    |    | -  |    |    |     |    |     | 43  |    |    |    | - |
|                                   |  |     |   |   |   |   |   |     |   |    |    |    |    |    |    |    |    |     | 9  | 118 |     |    |    |    |   |
|                                   | 434 L  |     |   |   |   |   |   |     |   |    |    |    |    |    |    |    |    |     |    |     |     |    |    |    |   |
|                                   |  |     |   |   |   |   |   |     |   |    |    |    |    |    |    |    |    |     |    |     |     |    |    |    | - |
|                                   |  |     |   | 3 |   |   |   |     |   |    |    |    |    |    |    |    |    |     |    |     |     |    |    |    | - |
|                                   |  |     |   |   |   |   |   |     |   |    |    |    | -  |    |    |    |    |     |    |     |     |    |    |    | - |
|                                   |  |     |   |   | 7 |   |   |     |   |    | 7  |    |    |    |    |    |    |     |    |     |     |    |    |    | - |
|                                   |  |     |   |   |   |   |   |     |   |    |    |    |    |    |    |    |    |     |    |     |     |    |    |    | - |
| MICROSON CONTRACTOR               | MES S  |     |   |   |   |   |   |     |   |    |    |    |    |    |    |    |    |     |    |     | 4.0 |    |    |    |   |
|                                   |  |     |   |   |   |   |   | X . |   |    |    |    |    |    |    |    |    |     |    | 134 |     |    |    |    |   |
|                                   | 200  | 5   |   |   |   |   |   |     |   |    |    |    |    |    |    |    |    |     |    |     |     |    |    |    |   |
|                                   |  |     |   |   |   |   |   |     |   |    |    |    |    |    |    |    |    |     |    |     |     |    |    |    |   |
|                                   |  |     |   |   |   |   |   |     |   |    |    |    |    |    |    |    |    |     |    |     |     |    |    |    |   |
|                                   |  |     |   |   |   |   |   |     |   |    |    |    |    |    |    |    |    |     |    |     |     |    |    |    | - |
|                                   |  | - 1 |   |   |   |   |   |     |   |    |    |    |    |    |    |    | 76 |     |    |     |     |    |    |    |   |
|                                   |  |     |   |   |   |   |   |     |   |    |    |    |    |    |    |    |    |     |    |     |     |    |    |    |   |
| Field Filtering Required Yes / No |  |     |   |   |   |   |   |     |   |    |    |    |    |    |    |    |    |     |    |     |     |    |    |    |   |

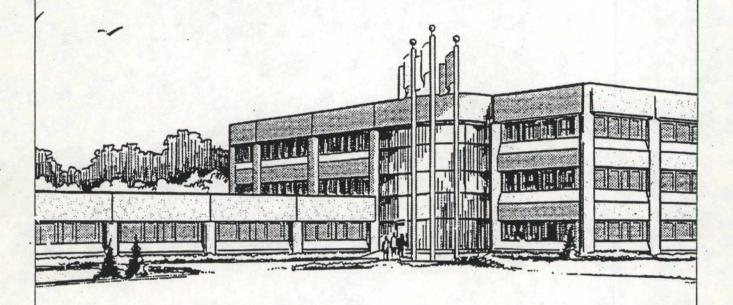
|          | NO.                      | DESCRIPTION                                  | PRESERVATIVE   | TAG COLOR             |
|----------|--------------------------|--|--|-----------------------|
|          |                          | Waters                                       |  |                       |
|          | 1                        | 40 ml Vial for Purgeable Organics            | 1+1 HCL Yes / No<br>Cool to 4° C                           | Yellow                |
|          | 2                        | 1000 ml Amber Glass Non Purgeable Organics   | Cool to 4° C   | Salmon                |
|          | 3                        | 125 250 500 1000 ml Plastic - Non Preserved  | Cool to 4° C   | Green                 |
|          | 4                        | 125 250 500 1000 ml Plastic - Nutrients      | pH < 2.0 w/H <sub>2</sub> SO <sub>4</sub>                  | Dark Blue             |
| _        | 5                        | 125 250 500 1000 ml Amber Plastic - Cyanides | pH to > 12 w/NaOH  | Light Blue            |
| MPLING   | 6                        | 125 250 500 1000 ml Plastic                  | pH to <2 w/HNO   | Red                   |
| 7        | 7                        | 1000 ml Glass - Oil & Grease / TPH           | pH to <2 w/H <sub>2</sub> SO <sub>4</sub>                  | Dark Blue             |
| 4        | 8                        | 125 ml Whirl Pac Bag / Bottle Bacteria       | Cool to 4° C   | White Label           |
| S        | 9 500 ml Glass - Sulfide |  | 0.5 ml Zinc Acetate<br>+ 0.5 ml NaOH to pH >9              | Light Green           |
| WATER    | 10                       | 250 ml Amber Glass - TOX                     | pH to < 2 w/H <sub>2</sub> SO <sub>4</sub><br>Cool to 4° C | Lilac                 |
| 3        | 11                       | 40 ml Amber Glass - TOC                      | pH to < 2 w/H <sub>2</sub> SO <sub>4</sub><br>Cool to 4° C | Pink                  |
|          | 12                       | 2000 ml Plastic - Radiological               | pH to < 2 w/HNO <sub>3</sub>                               | Gray                  |
|          | 13                       | 500 ml Amber Glass - Phenols                 | pH to < 2 w/H, SO,   | Brown                 |
|          | 14                       | 250 ml Amber Glass - Formaldehyde            | Cool to 4° C   | Orange                |
| 30       | 15                       | Other Water                                  |  |                       |
|          |                          | Soils / Non-Aqueous                          |  |                       |
| 9        | 16                       | 125 250 500 1000 ml Wide Mouth Plastic       | Cool to 4° C   | White                 |
| 5        | 17                       | 125 250 500 1000 ml Wide Mouth Amber Glass   | Cool to 4° C   | Manilla               |
| SAMPLING | 18                       | 125 ml Vial for Purgeable Organics in Soil   | Cool to 4° C   | Light Yellow          |
|          | 19                       | 125 ml Vial for TCLP Volatiles               | Cool to 4° C   | Yellow & Black Stripe |
| SOIL     | 20                       | 125 ml Wide Mouth Plastic - % Solids         | Cool to 4° C   | Yellow & White Stripe |
| 5        | 21                       | Other Soil / Non-Aqueous                     |  |                       |

## Appendix E

WWES Environmental Laboratory Quality Assurance/Quality Control Manual

## WW ENGINEERING & SCIENCE

Environmental Laboratory Division



Quality Assurance / Quality Control Procedures Manual

## WW ENGINEERING & SCIENCE ENVIRONMENTAL LABORATORY DIVISION QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES MANUAL

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## 1.0 PURPOSE OF THE MANUAL

### 1.0 PURPOSE OF THE MANUAL

The purpose of this manual is to specify procedures and technical requirements to be used by the WW Engineering & Science (WWES) environmental laboratories to assure that the data generated by the laboratory is accurate, reproducible and timely. This manual provides the chemistry laboratory a quality control plan which is to be used by every individual involved in the analytical efforts at WWES.

## 1.1 THE NEED FOR ANALYTICAL QUALITY CONTROL

There is a growing importance attached to the measurement of the concentration of any contaminant in water, effluents, and solid samples. As with any type of measurement the results of the methods utilized to measure the concentrations of these contaminants generally differ from the true concentration, i.e. all results are subject to error. Many experimental studies have shown that errors can arise which are as large as a 50 percent variations from the true value, and in fact, may vary between laboratories. Inaccurate analytical results restrict the ability of the analyst and the recipient of the data to draw valid conclusions and usually lead to false or misleading conclusions. Examples of common problems which arise during an analytical effort are as follows:

- A. Results, which are compared between two or more laboratories, are in error relative to each other.
- B. Results are to be used to decide if a water quality standard has been observed especially as the level of the analysis approaches the detection level.
- C. An inappropriate test procedure has been used to determine the analyte, resulting in values that do not represent the true sample concentration, i.e. direct aspiration of a turbid sample.

There is also increasing concern about the control of these errors being expressed at the local, the national and the international levels. The concern centers around the need to have a maximum amount of valid information obtained in a cost effective manner. In order to control errors, it is necessary to be able to measure the magnitude of these errors. This manual identifies the activities that are involved in the measurement and control of error. WWES considers analytical quality control of great importance, and requires that it be a primary feature in any analytical effort. The WWES requirements for analytical quality control are in concert with the quality control needs and demands of other organizations, such as the Environmental Protection Agency, various state regulatory agencies, and private industry.

Approximately twenty to thirty percent of all the available effort for routine analysis is absorbed in the execution of quality control requirements. It is often argued that the extent of this effort is too great with respect to routine laboratories and their need to be

profitable in their operation. The argument therefore claims that extensive quality control is an impractical expectation for a routine laboratory. However, the corporate policy at WWES demands that the appropriate level of quality control be applied to all analytical effort at WWES, regardless of the sample lot.

In the total effort, it is preferable to obtain twenty to thirty percent fewer results of known accuracy for each analytical batch than it is to obtain larger numbers of results of undefined accuracy. Due to the fact that all analytical procedures are subject to errors derived from many sources, it is not reasonable to assume that quality control is unnecessary with a "good" analyst. However, even a "good" analyst may not have an adequate idea of his (her) accuracy. Multiple studies by the EPA, both within laboratories and between laboratories, has shown this reasoning to be generally unsound.

# 2.0 QUALITY ASSURANCE ORGANIZATION AND RESPONSIBILITIES

## 2.0 QUALITY ASSURANCE ORGANIZATION AND RESPONSIBILITIES

## 2.1 OBJECTIVE OF THE QA PROGRAM AT WWES

The purpose of the quality control program is to continuously monitor error, both random and systematic, which inhibits the production of reliable and defensive analytical data. Error is inherent in any analytical routine, even with the most rigorous controls, and thus, a good QC program addresses not only the basic control techniques but also statistical means of measuring precision and accuracy and the confidence limits on these measurements.

The purpose of this manual is to specify the procedures, records, Chain-of-Command, and technical requirements which will be adhered to by the laboratories of WWES.

## 2.2 ORGANIZATION

Quality control at WWES begins with the bench analyst and moves up through the Chain of Command ultimately residing at the level of the President. A QC program which is administered only at the upper levels of management is doomed to failure and is unfair to the bench level analyst who needs a means by which he can observe the quality of his work. Quality Control is a two way program at WWES where directives from management are as important as suggestions and assistance from the bench analyst.

## 2.2.1 QC Chain of Command Flow Chart

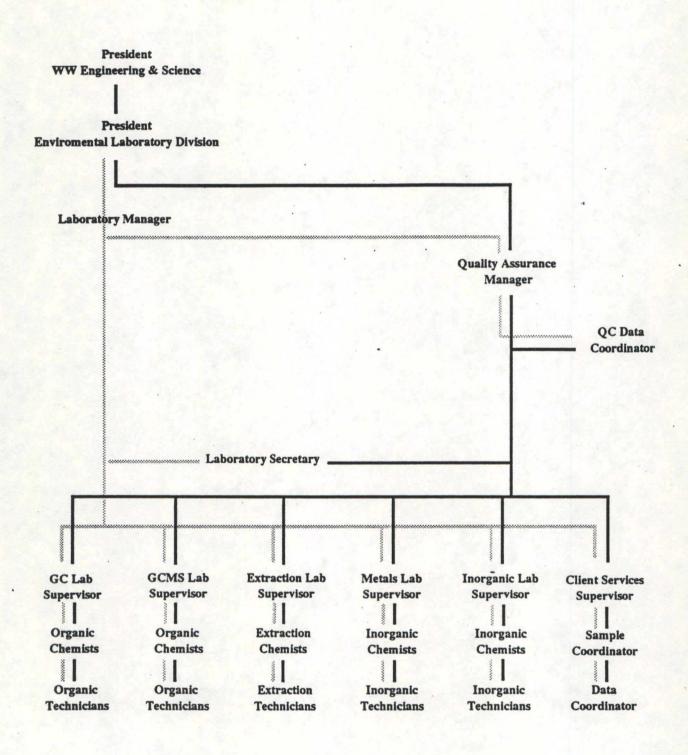
The following flow chart represents both the QA Chain of Command (solid line) and the Administrative Chain of Command (dotted). The flow chart represents the philosophy of WWES relative to the interaction of QC and production. Although the QC Manager reports to the Director of Analytical Services in the supervisory Chain-of-Command, his responsibilities for quality control require that he answer to the President of the Environmental Laboratory Division. The QC Manager acts as an immediate record keeper, QC administrator and liaison to the lab manager.

However, when a question relative to the quality of analytical data arises, the QA Manager, in conjunction with the President of the Environmental Laboratory Division, has the right to prevent data dissemination. In cases of conflict, the President of the Environmental Laboratory Division has final authority except when a compromise or directive is issued by the President of WW Engineering & Science.

## 2.2.2 Responsibilities of the Laboratory QC Manager

2.2.2.1 To monitor the Quality Assurance activities in the laboratory insuring adherence to all policies and procedures.

## QUALITY CONTROL CHAIN OF COMMAND FLOW CHART



Administrative Flow
Quality Control Flow

- 2.2.2.2 To identify problem areas and help in recommending improvement and changes.
- 2.2.2.3 To keep abreast of changing development in analytical QC particularly requirements set by regulatory agencies.
- 2.2.2.4 To arrange or produce random blind control samples.
- 2.2.2.5 To approve all laboratory data prior to recording such data for report generation purposes.
- 2.2.2.6 To maintain QC on all analytical activities and update control limits in a timely manner.
- 2.2.2.7 To oversee the maintenance of balance and controlled temperature apparatus record books on a daily basis and insure that such records are maintained on every piece of equipment.
- 2.2.2.8 To assure that bottle preparation, approval and storage meet established criteria.

## 2.2.3 Responsibilities of the Sample Coordinator

A full position description of the Sample Coordinator can be found in the "WWES Sample Receiving SOP".

- To insure that all samples received at WWES are properly preserved, split, logged-in, and stored in agreement with the log-in manual.
- To insure that all COC shipments are handled according to established procedures including storage, sample tracking and completion of files.
- To insure that all project sheets and subsequent paperwork is completed and filed.
- To insure that labile samples are distributed in a timely manner.
- To cooperate with the QC Manager in introducing blind samples.

## 2.2.4 Responsibilities of the Analytical Staff

- To insure that all records are generated and recorded on a daily basis.
- To insure that the following bench level QC requirements are met. To fill out lab notebooks daily as required. To provide for QC on every batch of samples. This level of effort generally includes:
  - 1) An initial calibration blanks and standards
  - 2) 10% sample matrix spikes

- 3) 10% sample matrix duplicates
- 4) Laboratory control sample and a method preparation blank for each batch.
- To insure that instruments are calibrated prior to initiating any analysis and that no analyses are started unless calibration has been satisfactorily completed.
- To insure that every batch of analyses meets established QC guidelines or is reanalyzed automatically.
- To inform the lab manager of any reoccurring problems or systematic trends which may effect quality.

## 2.2.5 Responsibilities of the Laboratory Supervisor

- To insure that sufficient competent staff is available to administer QC.
- To insure that all participating analysts are certified in the test they are performing.
- To insure that effective training and orientation takes place for every new analyst.
- To insure that all QC procedures, directives or project oriented requirements are met.
- To review all preliminary reports and approve them prior to the generation of a final report.
- To interface with the QC coordinator and QC Manager on a routine and consistent basis.
- To take responsibility for immediate solutions to QC problems which may slow or stop production.

## 2.2.6 Responsibilities of the Data Coordinator

The Data Coordinator's (DC) responsibilities are:

- To enter all data generated into the appropriate records.
- To insure that the Laboratory Supervisor has signed the data forms (bench sheets prior to entry).
- To inform the QC Manager when a project is complete and ready for a preliminary report.
- To provide corrections to all reports from preliminary report feedback.

## 3.0 FACILITIES AND EQUIPMENT

## 3.0 FACILITIES AND EQUIPMENT

- 3.1 The physical plant layout diagram is enclosed. The approximate square footage allocated to each analysis area is presented as well as the number of personnel normally working in each area. A listing of equipment presently utilized by WWES is also enclosed.
- 3.2 The quality of the analytical instrumentation utilized by WWES is of great importance considering its ultimate effect on data quality. The following guidelines exist for the procurement of analytical instrumentation:

## 3.2.1 Equipment Need

An equipment need is identified by the Lab Manager or the President as a result of:

- o New Contractural Effort
- o Regulatory Changes
- o Normal Upgrade/Replacement
- o Capacity Improvements

#### 3.2.2 Procurement Procedure

The performance specifications defined by the need are used to identify prospective equipment suppliers. The Lab Manager mails the performance specifications to the prospective equipment suppliers. Those suppliers able to meet the performance specifications are asked to provide a quotation for the purchase or lease of the equipment. An evaluation of the quotations is made by the Lab Manager with consideration given to such items as: equipment ease of use, degree of automation, specification compliance, potential for computerization, price and space requirements.

A written recommendation by the Lab Manager is presented to the President for their review and comment.

A final recommendation by the President of the Laboratory Division is made to the Vice President of Corporate Finance. The final approval is granted based on the assurance of complying with all regulatory and corporate guidelines for the generation of the highest quality data.

#### 3.3 CHEMICAL PROCUREMENT AND INVENTORY PROCEDURE

All reagent specifications are dictated by the EPA/APHA or NIOSH approve analytical methods. These reagent specifications are identified and maintained on the chemical inventory index card system. The chemical inventory system assures the order of chemical use and minimizes the possibility of exceeding their useful shelf life. The

addition of a new method or a change in an existing method that requires a corresponding addition or change in a reagent used for that method will be identified by the Lab Area Supervisor or Group Leader. The Supervisor or Group Leader will update the chemical inventory.

All reagent specifications including available vendors are listed in the chemical inventory tables.

All chemical reagents are received by a representative from the appropriate lab area. The representative opens the shipping package and compares the packing slip with the contents. Discrepancies are identified to the Area Supervisor. The materials receipt is identified and recorded on the chemical inventory. The materials are then inventoried on the chemical inventory index. The index system identifies the amount(s) received and when. When the last bottle/container of the chemical remains, the chemical is placed on an open order sheet located in the lab.

The group leader/supervisor for that lab area is responsible for picking up the chemical open order sheet each week and preparing a purchase order. The purchase order is approved by the Lab Manager and a typed purchase order is issued to the approved vendor that has been previously identified as being able to supply the specified material. The receipt of the new order initiates the inventory system activities.

#### 3.4 PREVENTATIVE MAINTENANCE

Every analytical instrument has a separate maintenance log book as identified in the Document Control Section No. 7.3.9. The required maintenance activities have been developed by the Lab Manager and each Group Leader/Area Supervisor. The maintenance activities comply with manufacturer specifications and working experience requirements.

Each maintenance log book contains a table indicating the frequency and type of maintenance required. The maintenance activity is documented each day or as the frequency requirements dictate.

Analysts are assigned the responsibility of maintaining various instruments or equipment in their respective laboratory areas. The Group Leader or Area Supervisor is responsible for checking the maintenance log books each week and signing off as checked. The Quality Assurance Manager is notified of any deviations or lack of maintenance activity performance, and corrective actions are taken.

## ANALYTICAL EQUIPMENT WWES-INORGANIC LABORATORY

| Instrument Description               | Manufacturer                    | Model No.  |
|--------------------------------------|---------------------------------|------------|
| Analytical Balance (2)               | Metler                          | AE163      |
| Analytical Balance                   | Metler                          | PC4400     |
| Analytical Balance                   | Metler                          | BB2400     |
| Auto-Analyzer (dual channel)         | Bran & Lubbe                    | TRAACS 800 |
| Auto-Analyzer                        | Technicon                       | AAII       |
| Auto-Analyzer                        | Lachat                          | Quick Chem |
| Conductivity Meter                   | YSI                             | 32         |
| FTIR Spectrophotometer               | Perkin-Elmer                    | 1600       |
| pH/mv Meter                          | Beckman/Altrex                  | 70         |
| pH/mw/ISE Meter                      | Orion                           | EA920      |
| Spectrophotometer (visible)          | Hitachi                         | 100-40     |
| Spectrophotometer (UV-VIS)           | Shimadzu                        | 1604       |
| Spectrophotometer (UV-VIS)           | Shimadzu                        | UV-1201    |
| Total Organic Carbon Analyzer (TOC)  | O.I.C.                          | 700        |
| Total Organic Halogen Analyzer (TOX) | Xertex/Dohrman                  | ***        |
| Nephlometer                          | НАСН                            | 2100A      |
| Polarograph                          | EG&G Princeton Applied Research | 384B       |
| Auto-Titrator                        | Metler                          | DL12       |

<sup>( )</sup> Designates multiple units.

## ANALYTICAL EQUIPMENT WWES-METALS LABORATORY

| Instrument Description             | Manufacturer | Model No.  |
|------------------------------------|--------------|------------|
| AA Spectrophotomer (flame)         | Perkin Elmer | 5000       |
| AA Spectrophotomer (flame/furnace) | Perkin Elmer | 5100 PC    |
| AA Spectrophotometer (furnace)     | Perkin Elmer | 5100 PC    |
| ICP Spectrophotometer              | Perkin Elmer | Plasma 40  |
| ICP Spectrophotometer              | Perkin Elmer | Plasma 400 |
| Autosampler                        | Perkin Elmer | AS-50      |
| Autosampler                        | Perkin Elmer | AS-51      |
| Autosampler                        | Perkin Elmer | AS-60      |
| Autosampler                        | Perkin Elmer | FIAS-200   |
| Mercury Analgam System             | Perkin Elmer |            |
| Microwave Digestion System         | CEM          | MDS 810    |
| Ultrasonic Nebulizer               | CTech        | •          |

## ANALYTICAL EQUIPMENT WWES-GC LABORATORY

| Instrument Description      | <u>Manufacturer</u> | Model No. |
|-----------------------------|---------------------|-----------|
| Gas Chromatographs (3)      | Varian              | 3700      |
| w/ECD (2), PID/FID, FID/ECD | ·                   | •         |
| Gas Chromatograph (3)       | Varian              | 3400      |
| w/FID/CED                   | • .                 |           |
| Gas Chromatograph (2)       | Tracor              | 540       |
| w/Hall-PID                  |                     |           |
| Gas Chromatograph (2)       | Tracor              | 585       |
| w/Hall-PID                  | •                   |           |
| Gas Chromatograph           | Tracor              | 9000      |
| w/Hall-PID                  |                     | • •       |
| Gas Chromatograph           | HNU .               | 301       |
| w/FID                       |                     | · ·       |
| Autosampler                 | Varian              | 8000      |
| Autosampler (3)             | Tekmar              | ALS 2016  |
| w/Auto Sample Heater        |                     |           |
| Concentrator (4)            | Tekmar              | LCS 2000  |
| Autosampler                 | Tekmar              | ALS 2050  |

<sup>( )</sup> Designates multiple units.

## ANALYTICAL EQUIPMENT WWES-GC LABORATORY

| Instrument Description         | <u>Manufacturer</u> | Model No.      |
|--------------------------------|---------------------|----------------|
| Autosampler (3)                | Tekmar              | ALS (10 place) |
| Concentrator (3)               | Tekmar              | LSC-2          |
| Thermal Tube Desorber          | Envirochem, Inc.    | 850            |
| HPLC (UV)                      | Isco                | 2300           |
| HPLC (UV-Fluorescence)         | Perkin Elmer        | Series 410     |
| LC Oven                        | Perkin Elmer        | 101            |
| Diode Acray Detector           | Perkin Elmer        | 235            |
| Fluorescence Detector          | Perkin Elmer        | LC240          |
| Chromatography Data Systems:   |                     |                |
| Integrators (7)                | Spectra Physics     |                |
| Turbochrom                     | Perkin Elmer        |                |
| ( ) Designates multiple units. |                     |                |

## ANALYTICAL EQUIPMENT WWES-GC/MS LABORATORY

| Instrument Description  | <u>Manufacturer</u> | Model No. |
|-------------------------|---------------------|-----------|
| Mass Spectrometer (2)   | Finnigan Mat        | OWA 1000  |
| Mass Spectrometer (2)   | Extrel              | ELQ-400   |
| Mass Spectrometer (2)   | Varian              | Saturn    |
| Autosampler (2)         | Varian              | 8000      |
| Gas Chromatograph (2)   | Varian              | 3400      |
| Gas Chromatograph (2)   | Perkin Elmer        | Sigma 3B  |
| Gas Chromatograph w/FID | Varian              | 3400      |
| Autosampler             | Tekmar              | ALS 2016  |
| Concentrator            | Tekmar              | LCS 2000  |
| Concentrator            | Tekmar              | LCS-2     |
| Concentrator            | Tekmar              | LCS-2000  |

<sup>()</sup> Designates Multiple Units

## COMPUTING EQUIPMENT WWES-ALL LABORATORY AREAS

| Instrument Description | <u>Manufacturer</u>     | Model No.      |
|------------------------|-------------------------|----------------|
| Mini-Computer          | Digital Equipment       | Micro-Vax II   |
| Personal Computer (18) | Club                    | 386            |
| Personal Computer (7)  | Club                    | 286            |
| Personal Computer (3)  | Everex Step             | 386            |
| Personal Computer (2)  | Macintosh               | Plus           |
| Personal Computer      | Macintosh               | SE             |
| Personal Computer (2)  | Compac                  | 386            |
| Personal Computer (2)  | Zenth                   | 386            |
| Personal Computer      | Epson                   | Equity III     |
| Printer (3)            | Pentronix               | 6280           |
| Printer (2)            | Hewlet Packard          | Laser Jet      |
| Printer                | Hewlet Packard          | Laser Jet II   |
| Printer                | Hewlet Packard          | Laser Jet IIID |
| Printer (6)            | Epson                   | FX-850         |
| Printer                | NEC-Laser Silent Writer | LC890          |

<sup>( )</sup> Designates multiple units.

## PHYSICAL PLANT:

Laboratory Name: Environmental Laboratory Division

WW Engineering & Science, Inc.

Address 5555 Glenwood Hills Parkway S.E.

Grand Rapids, MI 49588

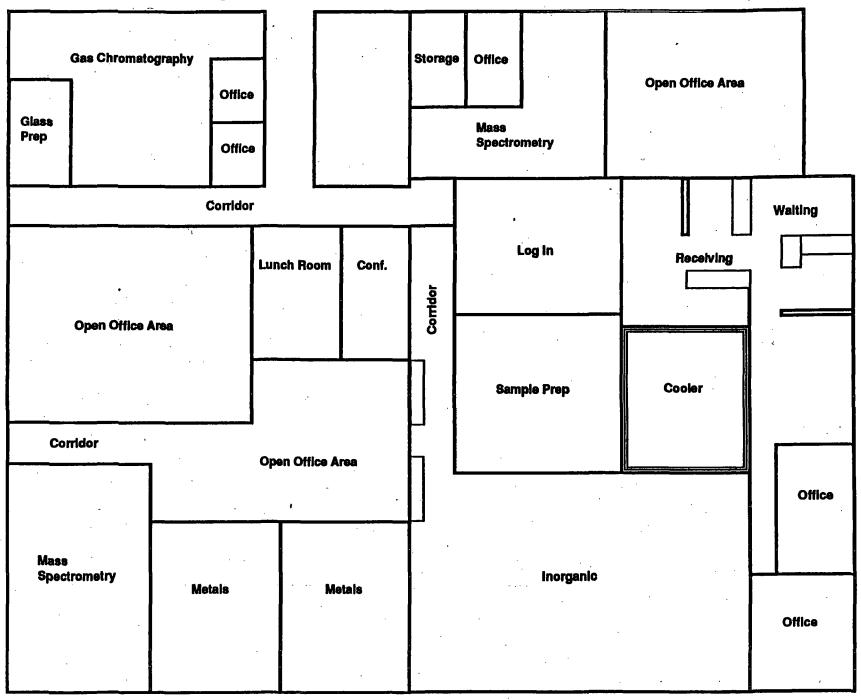
President Richard R. Rediske, Ph.D.

Vice President/Lab Manager John P. Dullaghan, MBA

An attached drawing of the laboratory indicates the general areas of analysis, the space allotted to each, and the number of personnel generally assigned to each area.

| Analysis                    | Space Allotted, Ft <sup>2</sup> | No. of<br>Personnel<br>(September, 1991) |
|-----------------------------|---------------------------------|--|
| Wet Chemistry/Microbiology  | Approx. 2000                    | 12                                       |
| Atomic Absorption/Emission  | Approx. 2000                    | . 12                                     |
| Gas Chromatography (GC)     | Approx. 2400                    | 10                                       |
| GC/Mass Spectrometry        | Approx. 1500                    | 8  |
| Sample Processing & Storage | Approx. 1500                    | 5  |
| Administrative Offices      | Approx. 2500                    | 20                                       |
| Organic Pretreatment        | Approx. 1000                    | 6  |
| Laboratory Offices          | Approx. 1000                    | 10                                       |

## **WW Engineering & Science Environmental Laboratory Division**



## 4.0 ANALYTICAL METHODOLOGIES

### 4.0 ANALYTICAL METHODOLOGIES

### 4.1 METHODS UTILIZED

The WWES Laboratory maintains and updated reference volumes of approved analytical methodologies for environmental and non-environmental analysis. A responsibility of the Lab Manager and the President of the Environmental Laboratory is to continually seek and review regulatory method changes and their impact on current laboratory practices. The most commonly referenced materials include:

- "Methods for Chemical Analysis of Water and Wastes" EPA-600/4-79-020 revised March, 1983.
- "Manual of Analytical Procedures" NIOSH, Volumes 1 & 2, Third Edition, Feb., 1984.
- Standard Methods for the Evaluation of Water and Wastewater 17th Edition, APHA, AWWA, WPCF; 1989..
- "Handbook for Analytical Quality Control in Water and Wastewater Laboratories", EPA 600/4-79-019, March 1979.
- "Physical and Chemical Methods for the Evaluation of Solid Waste" EPA-SW846
   Third Edition, 1990.
- "Guidelines Establishing Text Procedures for the Analysis of Pollutants". CFR July 1, 1990.

### 4.2 METHOD CALIBRATION AND OPERATING PROCEDURES

A standard operating procedure manual exists for all analytical procedures. The S.O.P's include specific calibration procedures that must be followed by an analyst prior to conducting sample analysis. The analyst is required to perform and document the calibration procedure. The calibration activity is identified by each analyst in their lab notebooks. The actual standards utilized are found in each instrument log book. It is the responsibility of each analyst to document all calibration and operating procedures utilized in the instrument log books. It is the responsibility of the group leaders and/or supervisors to notify the Quality Assurance Manager when deviations occur so that corrective actions can be taken. The corrective action will be to identify whether the information is simply missing (not entered) and to have it recorded or if the calibration has not been performed, to not release data generated that day and require those samples to be rerun.

mn-f:qa-qc

## 5.0 METHOD CERTIFICATION

#### 5.0 METHOD CERTIFICATION

All methods used by WWES which were not developed by WWES will be certified prior to their use. Method Certification is contiguous with the certification of the analyst and requires essentially the same analytical program. Method certification is necessary in order to establish detection limits, method application limits and criteria for control limits. In most cases, detection limits and recoveries stated in a method are obtained under ideal conditions and do not reflect real world solutions, i.e., silty well water and industrial effluent versus a drinking water supply. Method certification falls into 2 categories: 1) Methods being employed for the first time and 2) Methods which are to replace currently certified methods (replacement methods). In either case, analysis of client sample may not proceed until certification has occurred.

## 5.1 METHOD CERTIFICATION

## 5.1.1 Linear Range

The first step in certifying a method is to establish the linear range (operating range) of the method. A method may be used only over the range in which it is linear. Some methods do not have linear ranges but curves from which results are calculated. For the moment we will ignore methods with curves. A linear range must be established independent of the method data since instruments can effect the range. Standards and multiple detections will be used for establishing the linear range. For example, a range of 1 to 1000 has 3 decades (3 orders of magnitude or 103). Therefore, a range of 1 to 1000 requires 11 levels of test standards (.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, 1000). Notice that each decade follows the 0.5x to 10x rule, i.e. the area 10 to 100 is covered by 5,10,20,50 and 100. The range to be attempted is dependent on the method, the instrument and the analytical supervisor. If the responses show linearity, the range has been established. If, however, a curve develops or there appear to be two linear ranges, the standards must be repeated including additional levels to verify the status of the questionable area.

## 5.1.2 Working Curves

Some methods operate from a curve response, i.e. sodium by emission spectroscopy. The method will indicate the working curve which must be verified. The method with working curves requires a full curve each time an analysis is to be performed.

## 5.1.3 The Generation of the Method Detection Limit

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be identified, measured and reported with 99% confidence that

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the analyte concentration is greater than zero and determined from analysis of a sample in a given matrix containing analyte.

#### SCOPE AND APPLICATION

This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

The MDL obtained by this procedure is used to judge the significance of single measurement of a future sample.

The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device or instrument independent.

#### **PROCEDURE**

- 1. Make an estimate of the detection limit using one of the following:
  - (a) The concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5. if the criteria for qualitative identification of the analyte is based upon pattern recognition techniques, the least abundant signal necessary to achieve identification must be considered in making the estimate (PCB).
  - (b) The concentration value that corresponds to three times the standard deviation of replicate instrumental measurements for the analyte in reagent water.
  - (c) The concentration value that corresponds to the region of the standard curve where there is a significant change in sensitivity at low analyte concentrations i.e. a break in the slope of the standard curve.
  - (d) The concentration value that corresponds to known instrumental limitations.

It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the estimate of the detection limit.

- 2. Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferent concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.
- 3. (a) If the MDL is to be determined in reagent water (blank) prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated MDL (Recommend between 1 and 5 times the estimated MDL) Proceed to Step 4.
  - (b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated MDL proceed to Step 4.

If the measured level of analyte is greater than five times the estimated MDL, add a known amount of analyte to bring the concentration of analyte to between one and five times the MDL in the case where an interference is co-analyzed with the analyte.

If the measured level of analyte is greater than five times the estimated MDL there are two options:

- (1) Obtain another sample of lower level of analyte in same matrix if possible.
- (2) The sample may be used as is for determining the MDL if the analyte level does not exceed 20 times the MDL of the analyte in reagent water. The variance of the analytical method changes as in the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.
- 4. (a) Take a minimum of seven aliquots of the sample to be used to calculate the MDL and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If blank measurements are required to calculate the measured level of

analyte, obtain separate blank measurements for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.

- (b) It may be economically and technically desirable to evaluate the estimated MDL before proceeding with 4a. This will: (1) prevent repeating this entire procedure when the costs of analyses are high and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an incorrect MDL can be calculated from data obtained at many times the real MDL even though the background concentration of analyte is less than five times the calculated MDL. To insure that the estimate of the MDL is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower MDL. Take two aliquots of the sample to be used to calculate the MDL and process each through the entire method, including blank measurements as described above in 4a. Evaluate these data:
  - (1) If these measurements indicate the sample is in the desirable range for determining the MDL, take five additional aliquots and proceed. Use all seven measurements to calculate the MDL.
  - (2) If these measurements indicate the sample is not in the correct range, re-estimate the MDL, obtain new sample as in 3 and repeat either 4a or 4b.
- 5. Calculate the variance (S<sup>2</sup>) and standard deviation (S) of the replicate measurements, as follows:

$$S^{2} = \frac{1}{n-1} \left[ \sum_{i=1}^{n} \mathbb{X}_{i}^{2} - \left( \sum_{i=1}^{n} \mathbb{X}_{i} \right)^{-2} / n \right]$$

where the x1i = 1 to n are the analytical results in the final method reporting units obtained from the n sample aliquots and X,2 refers to the sum of the X values from i = 1 to n.

6. (a) Compute the MDL as follows:

$$MDL = t_{(n-1),-a} = .99$$
). S

where:

#### MDL - the method detection

t<sub>(n-1,1-a = .99)</sub> = the students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. See Table.

S = standard deviation of the replicate analyses.

(b) The 95% confidence limits for the MDL derived in 6a are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution (X2/df) and calculated as follows:

MDLlcl = 0.64 MDL MDLucl = 2.20 MDL

where MDLlcl and MDLucl are the lower and upper 95% confidence limits respectively based on seven aliquots.

- 7. Optional iterative procedure to verify the reasonableness of the estimated MDL and calculated MDL of subsequent MDL determinations.
  - (a) If this is the initial attempt to compute MDL based on the estimated MDL in Step 1, take the MDL as calculated in Step 6, spike in the matrix at the calculated MDL and proceed through the procedure starting with Step 4.
  - (b) If the current MDL determination is an iteration of the MDL procedure for which the spiking level does not permit qualitative identification, report the MDL as that concentration between the current spike level and the previous spike level which allows qualitative identification.
  - (c) If the current MDL determination is an iteration of the MDL procedure and the spiking level allows qualitative identification, use S2 from the current MDL calculation and S2 from the previous MDL calculation to compute the F ratio.

if  $S^2A/S^2B < 3.05$ 

then compute the pooled standard deviation by the following equation:

Spooled = 
$$\left[\frac{6S_A^2 + 6S_B^2}{12}\right] \frac{1}{12}$$

if  $S^2A/S^2B > 3.05$ , respike at the last calculated MDL and process the samples through the procedure starting with step 4.

(c) Use the Spooled as calculated in 7b to compute the final MDL according to the following equation:

$$MDL = 2.681$$
 (Spooled)

where 2.681 is equal to t(12,1-a=.99)

(d) The 95% confidence limits for MDL derived in 7c are computed according to the following equations derived from percentiles of the chi squared over degrees of freedom distribution.

where LCL and UCL are the lower and upper 95% confidence limits respectively based on 14 aliquots.

## REPORTING

The analytical method used must be specifically identified by number or title and the MDL for each analyte expressed in the appropriate method reporting units, if the analytical method permits options which affect the method detection limit these conditions must be specified with the MDL value. The sample matrix used to determine the MDL must also be identified with the MDL value. Report the mean analyte level with the MDL if a laboratory standard or a sample that contained a known amount analyte was used for this determination, report the mean recovery, and indicate if the MDL determination was iterated.

If the level of the analyte in the sample matrix exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL

#### REFERENCE

40 CFR Part 136 Appendix B, USEPA Chapter 1, 7/1/90.

Table of Students t Values at the 99 Percent Confidence Level

| Number of<br>Replicates | Degrees of Freedom (n-1) | t(n-1,1-oc=.99) |
|-------------------------|--------------------------|-----------------|
|                         | (                        | 2 142           |
| 7                       | 6                        | 3.143           |
| 8                       | 7.                       | 2.998           |
| 9.                      | 8                        | 2.896           |
| 10                      | 9                        | 2.821           |
| 11                      | 10                       | 2.764           |
| 16                      | 15                       | 2.602           |
| 21                      | 20                       | 2.528           |
| 26                      | 25                       | 2.485           |
| 31                      | 30                       | 2.457           |
| 61                      | 60                       | 2.390           |
| •                       |                          | 2.326           |

## 5.1.4 Method Spikes

Method spikes will be carried out over 2 separate days at the specified levels including blanks and calibration standards. The data obtained at the 2x or 5x level (for each certified range) will be used to establish a mean and standard deviation for initial control charts. Once this data has been generated and approved by the analytical manager, the method has preliminary certification and is ready for application to real world samples. These control limits will be updated with every batch of samples until 30 numbers have been developed to establish reliable control limits. After 30 data points the DC will provide updated control limits with each additional 20 numbers.

#### 5.2 REPLACEMENT METHOD CERTIFICATION

When a new method is to-be employed (where a new method is defined as including a new instrument method, i.e. flame vs flameless AA or Hall vs ECD), the method must be certified prior to its use on client samples.

Certification follows the procedures described in Sections 5.1. The results of these tests are important but are not necessarily compared to the current method. The detection limit may change and the work range may change but if they meet the needs of the lab, these changes are to be ignored. One may elect to utilize a t-test analysis to identify the method differences as being significant or not.

## 5.2.1 Comparison by the t-Test

One sample will be analyzed a minimum of 4 times by each method. The results will be subject to a t-test analyses. If the t-test indicates statistical correlation, regardless of the correlation coefficient, the new method is certified. If the t-test fails, refer to Section 5.2.2 below.

## 5.2.2 Decisions on Certification

The purpose of a new method is to improve accuracy, precision and efficiency. Efficiency is of no consequence if a method is imprecise and inaccurate, and therefore, is not a consideration in certifying new methods. However, a new method may fail the t-test because it is more accurate and/or more precise. Careful consideration and more analyses may be necessary with a new method by the analytical manager and supervisor.

## 6.0 ANALYST TRAINING AND CERTIFICATION

## 6.0 ANALYST TRAINING AND CERTIFICATION

## 6.1 RATIONAL

Consistent with requirements by the EPA and other regulatory agencies for analyst training and certification programs, WWES has a strict policy relative to the training and certification of analysts prior to their involvement in the analysis of client samples. The program is necessary in order to maintain continuity in all analytical programs and to insure the integrity of all data.

### 6.2 TRAINING

The supervisor is responsible for training all new personnel. This training will be in conjunction with the group (workstation) and group leader if applicable. Training will include, but not be limited to, WWES QC requirements, paperwork flow, lab safety and organizational structure. In addition, the new analyst will be given copies of the QC manual, log-in manual and methodologies which the analyst will be required to read. Training in the methods to be used will be initiated prior to analyst certification.

#### 6.3 · CERTIFICATION

Each new WWES analyst will be required to receive certification on all methods which he is to perform. Certification insures that the analyst can meet WWES detection limits and quality control limits as established for the method. Certification includes two parts, both of which must be completed satisfactorily.

### 6.3.1 Method Spikes

Analysis of spiked lab pure water at the levels of 0.5x, 1.0x, 2.0x, 5.0x and 10x where x is the established detection limit. This will include 2 blanks and a duplicated spike at 2.0x or 5.0x and will occur on 2 separate days. The data, where the duplicated results are averaged. These results must match current WWES Schwart control chart limits. Additional parameters such as consistent instrument calibration curves will be evaluated.

## 6.3.2 Check Sample Analysis

The analyst will test a known blind check sample in duplicate including a blank. All the data must fall within established control limits for the parameters.

### 6.3.3 Current Analysts Training

The LDI analyst, who is assigned a new method, must complete the certification program for the methods as outlined above prior to performing analyses on client samples.

#### 6.4 RECERTIFICATION

All WWES analysts will recertify on all their respective methods when required or demonstrated by two method spike performance failures following the procedures set forth in Section 6.3.1. The results must meet previous data, assuming that the same methods are employed.

#### 6.5 PERFORMANCE AUDITS

The Laboratory Manager, in cooperation with the QA Manager, will perform individual audits on all aspects of the operation biannually. These audits will include recertification data, control limits, all levels of records and laboratory performance on all check samples and instituted blind QC samples. A report of the audit results including recommendations will be forwarded to the President of the Environmental Laboratory Division.

# 7.0 DOCUMENT CONTROL, FLOW AND STORAGE

#### 7.0 DOCUMENT CONTROL, FLOW AND STORAGE

#### 7.1 PURPOSE

The paperwork trail must be designed to insure that after the issuance of a report, anyone - a client, a lawyer or the President of WWES can track a single sample result back through WWES records to the origin of the standards used in calibration and the identity of the person who prepared the sample bottles.

#### 7.2 PAPERWORK FLOW

As shown in the attached, "Flow Diagram" the paperwork trail is eventually the same for routine work as it is for samples under Chain-of-Custody. The general axiom is that a COC procedure is doomed to failure without a pre-existing scheme of tight sample and analytical control available as a routine measure. This contention, however, is only of minimal consequence with respect to the need for detailed records. The records trail can provide the following:

- Answers to questions of analytical integrity for results which are 2 months or two years old.
- Assistance in finding and solving random and systematic problems.
- Assistance in preventing long term degradation of analytical integrity.
- Assistance in insuring continuity of analytical effort despite personnel and mechanical changes.

# 7.3 DOCUMENT REQUIREMENTS

The following subsection identifies all documents which are generated during the course of any project:

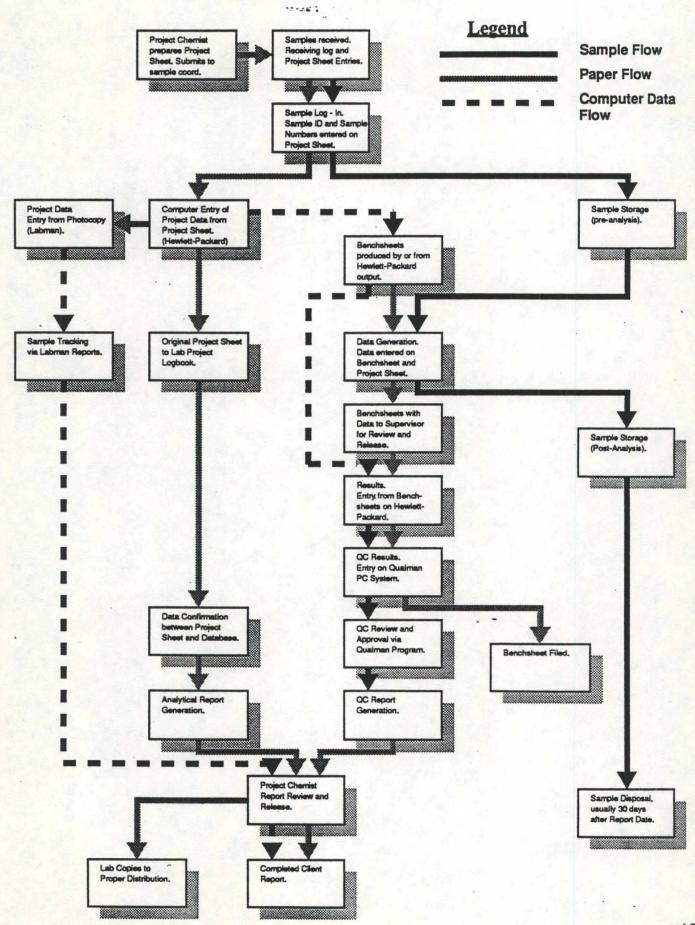
#### 7.3.1 Project Sheets

Every sample or group of samples which enter the WWES facility must be accompanied by the appropriate project sheet which has been properly filled out and provided to the Sample Coordinator (SC). The SC may not log-in samples for which there are not project sheets or for which there the project sheet is incomplete. An example project sheet is attached as Figure 2.

## 7.3.2 New Project Approval Form

Projects which require testing or analyses not routinely provided at WWES must have prior approval on a Project Questionnaire and commitment from the Analytical Manager and the head of the appropriate analytical group(s). For the project manager's purpose, the approval forms insure that the analytical testing

# Sample and Document Flow Diagram



# **ENVIRONMENTAL LABORATORY DIVISION Project Initiation**

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| Project Expiration                | Date                                   | <b>,</b> [ | 1 1                                     |     |              | 1                                      | 1        | Ш                                       |       | 1.         | ١     | -  |               |            |          |          | -                                       |  |  |            |            |   |            |          |   |                    | •    |
| Purchase Order No                 | lo. L                                  | <u></u>    |   |     | L            |  | 1_       | <u>    -</u>                            | _     |            |       |  |               |            |          | P        | roj.                                    | Ту   | oe (                                   | 1) [       | لــا       | F                                       | łер        | ort I    | Form                                    | nat <sup>(2)</sup> | لــا |
| Contract No.                      |  |            | لــــا                                  |     | 1_           | 1                                      |          | نــ                                     |       |            |       | ٠.   |               |            |          | F        | ield                                    | Bla  | ınk                                    | s L        |            | ٨                                       | feti       | nod      | s Pa                                    | ge                 | لــا |
| CCS Mgr.                          | لـــا                                  |            | 1_1                                     |     |              | 1                                      | 1        | Ш                                       |       |            | 1     |  |               | _L_        | ٦.       | С        | ase                                     | Na   | ırr.                                   | L          |            | C                                       | C          | Rep      | ort                                     |                    | ب    |
| CCS Project No.                   | لنا                                    | <u>_</u>   | 11                                      |     | ·<br>        | L                                      | L        |   |       |            |       |  |               |            |          |          |   |  |  |            |            |   |            |          |   |                    |      |

<sup>(1)</sup> C = Competitive Quote; D = Direct Request; R = Renewal (2) S = Standard; C = Cas# Report

| Frequency (                        | 1)   | Turnaround                            | لــــا      | Flame          |  | Bottles (4)  |            |
|------------------------------------|--|---------------------------------------|-------------|----------------|--|--|------------|
| Submit∕Yr.                         |  | C.O.C. (2)                            |             | Reactive       |  | Carrier  | <u> </u>   |
| # of Sample:                       | s LLLL   | QC Type (3)                           | نبيا        | Contact        | L  | Sample Storage                                     | لــــا     |
| -                                  |  |                                       |             | Health         | ليا  | Bottle Address (5)                                 | Ш          |
| (2) I = In<br>(3) RAS<br>(4) H = I | s between submittals<br>iternal (Lab)COC; E<br>, SAS, QAP<br>Hold; S = Ship<br>Yes if Bottle Address | = External (Fiek                      |             | •              |  | Narrative  | <u>.</u> . |
|                                    | Bottle   | Shipping Addres                       | SS          |                |  | Space for th                                       | ie.        |
|                                    |  |                                       |             |                | — .<br>— .                                   | Project Namis provided of the back side this sheet | ative and  |
|                                    | · · · · · · · · · · · · · · · · · · ·  |                                       | <u> </u>    |                | <del></del> .                                |  |            |
| Submitta                           | I Description  |                                       | /26 charact | ers available) | <u>                                     </u> | <u> </u>   |            |
| Date Exp                           | pected LLL-1   |                                       |             |                |  |  |            |
| Bottle Du                          | يا - لـــلــا Date   |                                       |             |                |  |  |            |
| Turnarou                           | and Days   |                                       |             |                |  | ·  | ٠          |
| Narrative                          | • <u> </u>   | ·                                     | ·           |                | •  |  |            |
| Submitta                           | al Narrative   |                                       |             |                |  |  |            |
|                                    |  | ·                                     |             |                |  |  |            |
|                                    |  |                                       |             |                |  | · · · · · · · · · · · · · · · · · · ·              |            |
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|                                    |  | · · · · · · · · · · · · · · · · · · · |             |                |  |  | ·          |
|                                    | •  |                                       |             |                |  | •  |            |

| Test Group Description | (40 characters available)             | Ш |                                       |
|------------------------|---------------------------------------|---|---------------------------------------|
| Sample Matrix          |                                       |   |                                       |
| Date Expected          | Bottle Due Date LL - LL               |   | J - L                                 |
| Parameters             | Method Number (Reference Citation)    |   | Specific DL                           |
|                        |                                       |   |                                       |
|                        | •                                     |   |                                       |
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area has received notification and will be prepared. For the analytical managers purpose, proper notification has been received and sufficient time has been allotted for preparation and development. Projects requiring rush turn around on modified methods must be approved as well. An example of a Project Questionnaire is attached as Figure 3.

# 7.3.3 Problem Project Sheets

When the Sample Coordinator (SC) identifies a problem with a sample shipment or project sheet, a Problem Project Sheet will be initialed and sent to the project manager for resolution. See Figure 4.

### 7.3.4 Chain-of-Custody Forms

There are three forms for Chain-of-Custody samples. All three forms must be properly completed and included in the project file for each and every COC project.

#### 7.3.4.1 COC SHIPPING RECORD

The shipping record must be received in the shipping container with every COC shipment. The form attached as Figure 5 is similar to the form used by the EPA. This form will be used by WWES field samplers and returned with the samples. Other forms of a similar nature may be used by other clients. However, the information required on the WWES form must be present on any other client form or they run the risk of their COC being rejected as a continuous trackable COC event.

#### 7.3.4.2 COC SAMPLE CONTROL RECORD

This form is used as a record of the movement of COC samples in and out of the COC locked storage. The analyst signs samples in and out each time a sample(s) is removed for any analysis. A copy of the form is attached as Figure 6. After all analyses are complete, the Sample Coordinator files the form in the COC project file.

# 7.3.5 Work Sheets/Project Sheets

Work sheets are the analytical assignment forms generated by the computer or the lab manager within 24 hours after log-in for each project or group of projects. The work sheets are divided into work stations, i.e. the analytes for which one or more analysts has sole responsibility. In many cases, the work sheets will have an entry position for the results of each analyses for each sample. In either case, the work sheet, upon completion of all analyses, will be turned into the appropriate

# ENVIRONMENTAL LABORATORY DIVISION PROJECT QUESTIONNAIRE

# REQUEST FOR WORK/QUOTATION (circle one)

| Client                                     | Proj. No.             |                   | _ `         |                                       |
|--|-----------------------|-------------------|-------------|---------------------------------------|
| Client Project Name                        | ·                     | Proj. Mgr.        |             | Initials:                             |
| HOW OO VOU WANT I                          | t to look on the redo | DFL)?             |             | .*                                    |
| Where should report Date of request of w   | t be routed?          | <del></del>       |             | <u> </u>                              |
| Date of request of w                       | ork?                  | Lab Notified      | YES         | NO                                    |
| Date samples will as Project Frequency:    | mive in lab:          | <del></del>       |             | <del> </del>                          |
| Project Frequency:                         | One Time              | Other (specify    | y)          | <del></del>                           |
| Turnaround required                        |                       |                   |             |                                       |
| Confirmed in ELD                           |                       | <u> </u>          | <del></del> | <del></del>                           |
| Job Description:                           |                       | 10.010            |             | _ <del></del>                         |
| Quality control requ                       |                       |                   |             |                                       |
| Does QC need to be                         |                       |                   | 10          |                                       |
| Is strict Laboratory                       |                       |                   | NO          |                                       |
| Have sample contain                        |                       |                   |             | •                                     |
| Sample containers f                        |                       | requested from    |             |                                       |
| Grand Rapids/Livor                         |                       |                   |             | •                                     |
| No. of water sample<br>Parameters required | es:                   | (circle es-):     |             |                                       |
| Parameters required                        | are or/see attached   | (circle one):     | <del></del> |                                       |
|  | <del> </del>          |                   |             | <del></del>                           |
|  |                       | _ <del></del>     |             | ···                                   |
|  |                       |                   |             | · · · · · · · · · · · · · · · · · · · |
|  | <u> </u>              |                   | <del></del> |                                       |
| Specific methods, d                        |                       |                   | · · ·       |                                       |
| No. of soil samples Parameters required    | •                     | (circle one):     |             | ·                                     |
|  |                       |                   |             |                                       |
|  |                       |                   |             |                                       |
|  |                       |                   | ·           | · · · · · · · · · · · · · · · · · · · |
|  |                       |                   |             |                                       |
| Specific methods, of 307, etc.)            | etection limits, and  | or program requir | rements     | (e.g. RCRA, Act                       |
|  | ·                     |                   |             |                                       |
|  |                       | <u> </u>          |             | <del></del>                           |
|  |                       |                   |             |                                       |
|  |                       |                   |             |                                       |
| No. of air samples                         |                       |                   |             |                                       |

|     | Specific methods, detection limits, and/or program requirements (e.g. ACGI   |
|-----|--|
|     |  |
|     |  |
|     |  |
|     |  |
|     |  |
| ļ   | No. of other samples: Type:  |
| F   | Parameters required are or/see attached (circle one):  |
| -   |  |
| -   |  |
| _   |  |
| _   |  |
|     | Specific methods, detection limits, and/or program requirements (e.g. Act 30 RCRA, etc.)   |
| -   |  |
| _   |  |
| _   |  |
| F   | Hazard levels associated with the samples are:   |
| =   |  |
|     | Has the client has been advised that any hazardous samples will be returned hem? YES NO  |
| I   | nem? YES NO Disposal of samples will take place 21 to 30 days after report mailing unless noted otherwise (If otherwise is noted a charge of \$5/sa nonth will apply). |
| (   | Costs for the analysis were confirmed by (ELD) of the Grapids Branch.  |
| I   | is there any particular format needed for the final report? YES NO (If yes discuss with ELD Project Chemist)   |
| A   | Are there any field measurements to be reported? YES NO If so specify  |
| _   | Are you running field blanks? YES NO   |
| 1   |  |
| . / | Are you running trip blanks? YES NO Other Information:   |
| . 1 | Are you running trip blanks? YES NO  |

# FIGURE 4

# WWES LABORATORY PROBLEM PROJECT REPORT

| SAMPLES REC   | EIVED ON AT AM/PM FROM:                             |
|---------------|---|
| AND DESCRIB   | ED AS WERE RECEIVED HAVING THE FOLLOWING .          |
| <del></del> . | WWES PROJECT APPROVAL FORM - ABSENT/INCOMPLETE      |
|               | CHAIN-OF-CUSTODY - ABSENT/INCOMPLETE                |
|               | CHAIN-OF-CUSTODY - DOES NOT MATCH SAMPLE TAGS       |
| <del></del>   | SAMPLE BOTTLES - BROKEN                             |
|               | SAMPLES ABSENT - QUAN. DOES NOT MATCH APPROVAL FORM |
| <del></del> . | SAMPLE BOTTLES - INCORRECT FOR ANALYSIS             |
|               | SAMPLE PRESERVATIVES - INCORRECT FOR ANALYSIS       |
|               | SAMPLE VOLUMES - INCORRECT FOR ANALYSIS             |
| <u> </u>      | SAMPLE TAGS - WRONG I.D./ABSENT                     |
|               | FIELD FORMS - ABSENT/INCOMPLETE                     |
|               | CUSTODY SEALS - ABSENT/NOT INTACT                   |
| _             | NON-ROUTINE PROJECT - NO PRIOR APPROVAL             |
|               | IN QUESTION WILL BE PROCESSED AS IS PLACED ON HOLD  |

THANK YOU
WWES LABORATORY
SAMPLE COORDINATOR

| WW Er      | iginer<br>Enviro | ering & S<br>nmental Lab<br>lienwood Hille Pr<br>and Aspida, Micr | cler<br>orato | ice,<br>ry Div | Inc.     |     | T      |         | Cha | ain c | of Cust              | ody Rec                 | ord                    |             | Nº 27                    | 823       |
|------------|------------------|---|---------------|----------------|----------|-----|--------|---------|-----|-------|----------------------|-------------------------|------------------------|-------------|--------------------------|-----------|
| Project No | 5.               | Project Nan   | 10            |                |          |     |        |         |     |       |                      | lype                    | Ę.                     |             |                          |           |
| Samplers   | (sign            | ature)  |               |                |          |     |        |         |     |       | No. of<br>Containers | Container Type & Volume | Preservation<br>Method |             | Analysis Required/Comm   | ents      |
| Date       | Time             | Matrix*   | Comp          | GRAB           |          |     | Sam    | ple I.C | ).  |       | 28                   | S = 4                   | P. S.                  |             |                          |           |
|            |                  |   |               |                |          |     |        |         |     |       |                      |                         |                        |             |                          |           |
|            |                  |   |               |                |          |     |        |         |     |       |                      |                         |                        |             |                          |           |
|            |                  |   |               | 1              |          |     |        |         |     |       |                      |                         |                        |             |                          |           |
|            |                  |   | - 69          |                |          |     |        |         |     |       |                      |                         |                        |             |                          |           |
|            |                  |   |               |                |          |     |        |         |     |       |                      |                         |                        |             |                          |           |
|            |                  |   |               |                |          |     |        |         |     |       |                      |                         |                        |             |                          |           |
|            |                  |   |               |                |          |     |        |         |     |       |                      |                         |                        |             |                          |           |
|            |                  |   |               |                |          |     |        |         |     |       |                      |                         |                        |             |                          |           |
|            |                  |   |               |                |          |     |        |         |     |       |                      |                         |                        |             |                          |           |
|            |                  |   |               |                |          |     |        |         |     |       |                      |                         |                        |             |                          |           |
| Relinquish | ed by:           | (signature  | 9)            | Date           | e / Time | •   | Receiv | red by  |     |       | Relinquis            | hed by:                 |                        | Date / Time | Received by: (signature) |           |
| Dispatched | by:              | (signature  | 9)            | Date           | e / Time | • ( | Carrie | r:      |     |       | Received             | to lab by:              |                        | Date / Time | Logged in by:            | Date/Time |

<sup>\*</sup> MATRIX: WATER (WTR), WASTEWATER (WW), SOIL (SOL), SLUDGE (SLU), AIR, OIL, HAZARDOUS WASTE (HW)

| 16-AUG-1 <sup>c</sup>    |  | WWES/ENVIRONMEN<br>CHAI   |   |                             | PAG  |
|--------------------------|--|---|---|-----------------------------|--|
| CLIENT:                  | Rumpke of Ohio, Inc.   | PROJECT:  | Quarterly Monitoring<br>Cincinnati, Ohio Landfill   | ·                           |  |
| BUBMITTAL:<br>PARAMETER: | August, 1991 Groundwaters<br>CARBON, TOTAL ORGANIC   | METHOD:   | TOC/OXID/WTR  | •                           |  |
| SAMPLE #                 | REMOVED BY:<br>(SIGNATURE)   | DATE & TIME<br>REMOVED  | RELINQUISHED BY:<br>DATE & TIME   | RECEIVED BY:<br>DATE & TIME | DATE & TIME<br>RETURNED  |
| 1279                     | and the case of th |   | بين مين القبل |                             |  |
| 1280                     | ally again any 1. Prince year man, man man hap year man apar any opin any man ally olive and viter and   | gape man sper gap your you, may have supplied upon the gap time tops distinct and | Gift parts year wind have you spin to the place while they die from spin wind will                            |                             | 000 PM 600 das vida vida vida con rida con cida con cida con |
| 1.281                    |  |   |   |                             |  |
| 1282                     | and a case of the state part and case case many specialists again and uniquied and think the state and   | ومن مثلة من الله من ديد بني وي من من وي وي وي وي وي وي وي وي                      |   |                             |  |
| 1283                     |  |   | and the same will be for had the spin per the day the had been and the same and                               |                             |  |
| 1201                     |  | •   |   |                             |  |

1285

Figure 6

supervisor with the proper bench sheets attached. Unless specifically advised, data will not be accepted on any form other than the project approval form sheets.

#### 7.3.6 Bench Sheets

The analysis of every analyte or group of analytes needed, i.e. VOA's requires a specific bench sheet which includes all results from the analysis of a group of samples. There are specific bench sheets for each analyte including specific requirements for their use. Examples of each bench sheet, can be found in Figures 7, 8 and 9.

#### 7.3.7 Lab Notebooks

The lab notebooks are the daily records of all activities of an analyst, or group of analysts, working in the lab. The notebooks will be bound and paginated. The notebook will be cleanly labeled on the inside cover with the date issued, the analyst's name, and the date completed. There are several specific rules which will be follows:

- All entries are in ink
- · There are no erasures, obliterations, or white outs allowed
- Corrections are single lined and initialed
- A new page is started each day or with every batch of samples
- Empty space is covered with a Z and signed and dated across the obtuse line
- Any and all work, observations and errors are noted
- Problem areas identified

When the instrument has just been repaired, a lamp changed, new column installed, detector repaired, or changed in any other manner, the log will also contain:

- A comment relative to the change or repair
- Reference page number to the Instrument Maintenance Log

The organic log books will also contain the following information relative to GC and GCMS oven and column conditions UNLESS they are exactly as specified in the referenced method which then will be commented on as such:

- column used (packing, diameter, length, type) o capillary as split or splitless
- · current type and flow
- make up flow if appropriate
- oven temperature and program if appropriate
- injector temperature
- detector temperature
- ion chamber voltage (GCMS)

| Test #: 198.0<br>Parameter: COPPER<br>Method: FLAME/<br>Ref. Cit.: USEPA- | TOTAL      | On<br>Un | DDL: 0.01<br>it: mg/l |                     |                    |               |                  | Benchs<br>C<br>Revi | rument #:<br>sheet ID:<br>Owner:<br>late run:<br>lewed By:<br>inal hrs: |                    |                                   |          |          |
|---|------------|----------|-----------------------|---------------------|--------------------|---------------|------------------|---------------------|---|--------------------|-----------------------------------|----------|----------|
| Comments:   |            |          |                       |                     |                    |               |                  |                     |   | Act a<br>Samples i | nal hrs:<br>in batch:<br>k std #: | 8        |          |
| Client<br>Submittal Sam   | ple COC    | QC.      | Reported<br>Conc.     | Duplicate<br>Result | Spike<br>Result    | Spike<br>Qty. | Spike<br>Stock # | % dif               | % réc   | ODL                | Analyst                           | EX       | C DNIR   |
| ICB:  |            | ¦        |                       |                     |                    | - XXXXXXXX    | XXXXXXXX         |                     |   |                    |                                   | -        | -        |
| ICV: Stk<br>GM BOC Lansing<br>453- 1 1                                    | <br>720    | <br>RAS  |                       |                     |                    | -             |                  |                     |   |                    | <br>                              | -        | _ii      |
| GM BOC Lansing<br>453- 1 1  |            | RASI     |                       |                     |                    |               |                  |                     |   |                    |                                   | _        |          |
| GM BOC Lansing<br>453- 1 1  |            | RAS      |                       |                     |                    |               |                  |                     |   |                    |                                   | -1<br>-1 |          |
| GM BOC Lansing<br>453- 1 1  | .723       | RAS      | j                     |                     |                    |               |                  |                     | !   |                    |                                   | _        |          |
| GM BOC Lansing<br>453- 1 1  | 724        | RAS      |                       |                     |                    |               |                  |                     |   |                    |                                   |          |          |
| GM BOC Lansing<br>453- 1 1  | 726        | RAS      |                       |                     |                    |               |                  |                     |   | <u> </u>           |                                   | _        |          |
| GM BOC Lansing 453- 1 1   | .727       | RAS      |                       |                     |                    |               |                  |                     |   |                    |                                   | _        |          |
| GH BOC Lansing 453- 1 1   | 728        | RAS      |                       |                     |                    | _i            |                  | İ                   | i   |                    | İ                                 | _        |          |
|   | <i>c</i> . |          |                       |                     |                    |               | , .              |                     |   |                    |                                   | ,        |          |
| MPB:  |            |          |                       |                     |                    | XXXXXXXX      | XXXXXXXX         |                     |   |                    |                                   |          |          |
| Digested LCS: Stk   | ·          |          |                       |                     | م مرحم من الأحاظ م |               |                  |                     |   |                    |                                   |          | <u> </u> |
| LCS: Stk  |            |          |                       |                     |                    |               | 0000000          |                     |   |                    |                                   |          | _ ! !    |
| CCB:  |            | _        |                       |                     |                    | XXXXXXXX      | XXXXXXX          |                     |   |                    |                                   |          |          |
| CCV: Stk  |            |          |                       | .!                  | <br>               |               | i<br>!           | <br>                | İ   |                    | <br>                              | _        | _        |

METALS BENCHSHEET

PAGE 1

| Test#: 389.01- 245.01  |  |                |                   |                            |  |         |                   |
|--|--|----------------|-------------------|----------------------------|--|---------|-------------------|
| Parameter: CHLORIDE Method: CL/TRAACS/WTR Ref. Cit.: USEPA-325.2  Comments:  | ODL:<br>Unit:                          | 2. 0<br>mg/1   |                   | 1.<br>2.<br>3.<br>4.<br>5. |  | OBS VAL | WKG STD<br>NUMBER |
| ClientSubmittal Sample COC   | ac #                                   | t/dil<br>actor | Reported<br>Conc. | ODL.                       | Spike<br>Qty<br>!XXXXXXX<br>!XXXXXXX         |         | EXC DNR           |
| ICV: Stk Rumpke of Indiana, Inc. 231- 7 1701 YES Rumpke of Indiana, Inc. 231- 7 1702 YES Rumpke of Indiana, Inc. 231- 7 1703 YES Nor-Am Chemical Co. 411- 1 1368 Nor-Am Chemical Co. 411- 1 1369 Nor-Am Chemical Co. 411- 1 1370 Nor-Am Chemical Co. 411- 1 1371 Nor-Am Chemical Co. 411- 1 1371 Nor-Am Chemical Co. 411- 1 1371 | RAS<br>RAS<br>RAS<br>RAS<br>RAS<br>RAS |                |                   |                            |  |         |                   |
| MPB:   | i                                      |                |                   |                            | XXXXXXXX<br>XXXXXXXX                         |         |                   |
| LCS:       Stk         SPK:       Stk         DUP:       Smp         DCB:       CCU:   |  |                |                   |                            | XXXXXXXX<br>XXXXXXXX<br>XXXXXXXX<br>XXXXXXXX |         | <br><br>          |

WWES/ENVIRONMENTAL LABORATORY DIVISION

Instrument #:
Benchsheet ID:
Owner:
Date run:
Supervisor:
Est anal hrs:
Act anal hrs:
Samples in batch:
Stock std #:
Uavelength (nm):
Cell path (mm):

PAGE 1

#### 7.3.9 Instrument Maintenance Log

The instrument maintenance log is a bound and paginated log which is used to track potential maintenance problems. The log is used every time the instrument is used but may contain several entries on one page. Entries on days where calibrations are correct may be as simple as "calibration met requirements". Anytime the instrument is repaired or modified in any way, the event must be noted with all specifics, including what was done, by whom, and why. A two detector GC has one log tracking, two detectors.

#### 7.3.10 Oven, Refrigerator and Freezer Temperature Logs

Each oven, insulator or furnace, plus all cold storage devices, will have their temperatures checked and recorded daily, or at a minimum, 5 days a week. Each device will have a thermometer in place or a temperature recorder in-place which will be checked by the Data Coordinator. A bound log book with 31 entries will be used to record all entries for each device upon which the DC will record the date and temperature and will initial the entry. The DC will have an NBS thermometer which will move between devices to act as a QC check for the primary temperature device. The log will include the second temperature when measured monthly.

# 7.3.11 Balance Logs

An Area Analyst will check all balances in the laboratory every day (or at least 5 days a week) using NBS class S weights. The analyst will record each day's reading in a log developed to handle every balance. A balance which fails to meet criteria will be removed from service until repaired. The DC will insure that every balance is serviced and calibrated annually recording such service in the log.

#### 7.3.12 Standard Record Books

Every standard used in the laboratory must be labeled and the label will possess the following information:

- The analyte or analytes contained in the standard
- The concentration
- The solvent
- The preservative, i.e. nitric acid
- The date made
- The Standard Reference Number

The last item, Standard Reference Number, is the identified standard and dilution sequence no. taken from the Standard Record Book in which the standard solution data is recorded.

All standards (including dilutions) will be recorded in a Standard Record Book assigned to the work station. Two record books will be used, each of which has a different purpose. The record books are subtitled as follows:

#### 7.3.12.1 STOCK STANDARDS LOG

This book contains standards starting with the identification of the starting material. One standard and/or standard mix with it's corresponding dilutions are identified.

#### 7.3.12.2 WORKING STANDARDS LOG

A working standard reference number is assigned and the corresponding dilutions are identified.

#### 7.3.13 Control Charts

Each analytical method will require at least one control chart. Some tests may involve several control charts, i.e. duplicate, matrix spikes and method spikes. The QC coordinator will supply the limits to be used to the work station involved. Every data point generated with every analytical batch will be plotted on the chart. Every out-of-control data point will be noted and an action indicated as to the disposition of the data. Completed control charts will be turned in to the DC for permanent change.

#### 7.3.14 Preliminary Reports

After all data has been entered for a project, the computer will flag a project ready for a preliminary report. The report will be identical to the final report in content except for the following:

- Preliminary Report will be reviewed and corrected if necessary on each page in large type.
- Comments necessary to the project will be printed under each sample or at the end of the report.

The DC will print the preliminary report and issue a copy along with the project file to the lab supervisor for review and corrections. The supervisor will sign off on the preliminary report after including comments, if appropriate, indicating that corrections are necessary. Afterwards, the supervisor(s) will pass the preliminary report to the QC Supervisor (QC) who will review and correct the report including a signature and comment. The QC will return the preliminary report

and file to the DC. The DC will make all corrections as required and review report structure for completeness. If no corrections are required, the DC will sign and date the preliminary report and place it in the Project File. The DC will then print a Final Report. When corrections are necessary, the DC will execute all corrections and indicate such changes on the initial preliminary, which is then filed in the project file. A new preliminary is then printed and issued for review.

#### 7.3.15 Final Report

After the preliminary report has been corrected and cleared all reviews, the DC will manually alter the computer flag and print a Final Report which will be placed in the project file folder and forwarded to the AM for approval. Space will be provided on the c.o.c. project file folder for the signatures of the Analytical Manager, the QA Manager and the Project Manager, all of whom are thus certifying that the report is complete, correct and defensible. The DC will then arrange for delivery of the final report.

#### 7.3.16 Project Files

The Project File is the comprehensive record of every project completed at WWES. A project file initially consists of a file folder set up by the Lab Secretary (LC) at the time of log-in. Chain-of-Custody projects will be stored in a locked COC file with strict limited access while routine project files are stored in a separate nominally limited access file. The LS will be responsible for including the following in the project file:

- Project Sheets
- Project Approval Sheets (if applicable)
- Problem Project Sheets
- Chain-of-Custody Forms
- All correspondence or documents received with the samples
- Preliminary Reports
- Separate Report Papers, i.e. Field Reports (if applicable) Final Report
- Any additional paperwork which may follow the report

All project files are stored for a period of 4 years.

# 8.0 SAMPLE CONTROL, FLOW, AND STORAGE

# 8.0 SAMPLE CONTROL, FLOW, AND STORAGE

All samples received at the WWES Engineering and Sciences must be logged in before any work is performed on the samples. This procedural requirement is specific not only to the chemistry lab, but the microbiological laboratory. The purpose of the log-in procedure, including sequential numbers assigned to all samples received in the facility, is to insure that WWES has a means by which samples can be tracked, data can be stored, and quality control can be tracked for any sequence of events during a particular analytical period. In handling projects in this manner, WWES, or the client, can insure a consistent and documented sequence of events under any analytical situation.

Management acknowledges that there are situations in which log-in of samples will be difficult due to rapid turn around requirements for particular compounds that may decompose or volatilize. An example of this kind of analysis is the total coliform samples which can be anticipated and for which holding times are short. The project approval form discussed within this manual will make it possible to preassign project numbers to samples arriving at the facility. Should a secondary mode of operation be necessary for the receipt of such samples, a mechanism will be developed between the sample coordinator and the Quality Assurance Supervisor. Any deviation from the standard log-in procedures detailed herein will be at the discretion of the laboratory supervisor or the laboratory manager. The execution of the log-in procedures for Chain-of-custody samples (see Section 8.8) is extremely crucial. Samples, that have been designated for Chain-of-Custody by a client, possess the potential of involvement in litigation or other legal situations., i.e. standards development or patents. By breaking Chain-of-Custody requirements, all results are invalid for such purposes.

#### 8.1 PROJECT INFORMATION

All information relative to a specific project must be recorded on a project approved form by the manager responsible for that project prior to the receipt and log-in of samples. Projects, and therefore samples which are not routine to the WWES laboratory, must have prior approval via the New Project Approval Form before samples may be received.

#### 8.2 NEW PROJECT APPROVAL

The project approval form include the following information:

- Client name, address, and client contact personnel
- Anticipated due date of the report (i.e. report in client hands by \_\_\_\_\_
- · Compound names or computer test codes or group computer test code
- Project and sample comments
- Contract number or purchase order for project
- Instructions relative to the proper completion of the project
- Pricing information relative to the proper completion of the project
- Chain-of-custody requirements

- Specific report requirements
- · Additional requirements such as rush, hazardous, labile

#### 8.3 NEW PROJECT APPROVAL

If a new project will require support from the analytical facilities, that project must be approved by the laboratory supervisors and the laboratory manager prior to project pricing and sample receipt. Routine samples are those samples and analyses which are continuously processed by WWES, such as priority pollutant samples, microbiological samples, and drinking water samples.

Projects which are non-routine are those that may require special testing, or which request parameters not routinely run within the laboratory, special holding times, or rush turn around. Non-routine projects will require that a New Project Approval Form be completed which includes the signatures of all the parties involved with the project. For example, if specific physical testing is necessary, the supervisor of the physical testing facility and the laboratory manager will have to sign off on the form thereby agreeing, not only to the project content, but for the turn around, the report requirements, the detection limits and the quality control reports that may be necessary to properly carry out the project requirements. Projects and/or samples arriving at WWES which are non-routine in nature, and for which there is no signed Project Approval For,, will not be processed. In this case, the manager responsible for the non-routine project will be advised of the problem and will then explain to the client why the delay is necessary for the execution of testing before proceeding to obtain the necessary approvals. The Project Approval Form must be completed and signed by all parties prior to the start of log-in.

#### 8.4 SAMPLE RECEIPT

#### 8.4.1 Introduction

All samples will be received at the WWES facilities by the Sample Coordinator (SC). The job description for the Sample Coordinator is attached as Figure 10. It will be the responsibility of the SC to determine: a) whether or not the proper project sheet is available for the arriving samples; b) whether or not the samples require chain of custody; c) whether or not the samples are labile in nature and require immediate attention; d) the manner in which those samples will be split, preserved and stored or routed. It is the objective of the SC to insure that the receipt of all samples is consistent with the requirements of the WWES Manual and that all pertinent information relative to those samples is recorded. This information may be used in client reports, communicated to the laboratory or to the client and, in some cases, reported to a legal authority relative to Chain-of-Custody samples.

#### FIGURE 10

#### SPECIFIC RESPONSIBILITIES

The SC's duties and responsibilities shall include, but not be limited to:

- 1. Sample receipt.
- 2. Insuring that COC sample receipt includes shipper's signature on COC forms.
- 3. Inspection of sample shipping containers for presence/absence and condition of:
  - a) custody seals, locks, "evidence tape", etc.
  - b) container breakage and/or container integrity
- 4. Recording conditions of both shipping containers and sample containers (bottles, jars, cans, etc.) in appropriate logbooks or on appropriate forms.
- 5. Signing appropriate documents shipped with samples (i.e., Chain-of-custody record(s).
- 6. Verifying and recording agreement or non-agreement of information on sample documents (i.e., separate tags, Chain-of-Custody records, traffic reports, airbills, etc.) on appropriate forms and on the WWES project sheet.
- 7. Initiating the sample and project log-in procedures on appropriate laboratory documents and according to the WWES Log-in Procedures document, including the initiation of project files with sample control records.
- 8. Marking or labeling samples with laboratory sample numbers, as appropriate.
- 9. Placing samples and spent samples into appropriate storage and/or secure areas.
- 10. Controlling access to samples in storage and assuring that laboratory operating procedures are followed when samples are removed from and returned to storage.
- 11. Monitoring storage conditions for proper sample preservation such as refrigeration temperature and prevention of cross-contamination.
- 12. Returning shipping containers to the proper client or licensed disposal facility.
- 13. Providing for the splitting of samples into required aliquots, including preservation for each working station.

# 8.4.2 Examination of Shipping Container

Immediately upon receipt of a sample shipment at WWES, the SC will examine the shipping container (the container may be a box, a cooler, a styrofoam container, etc.) to ascertain and document the condition of the samples and to process Chain-of-Custody papers, where appropriate. The SC will record the condition of the shipping container, the identification of the shipper, the presence or absence of any seals on the container (if it is Chain-of-Custody), and the labeling which may include special instructions prior to opening the container. If the shipping container is damaged, a report will be sent immediately to the shipper and the lab supervisor (see Section 8.15.2, Problem Project Sheet).

## 8.4.3 Carrier Sign Off for Chain-of-Custody Container

Should the SC identify the shipping container as being a Chain-of-Custody container, the SC will attempt to have the carrier's representative sign off on the Chain-of-Custody papers which should be available either on the outside of the shipping container, or immediately inside. An example of a Chain-of-Custody record is attached as Figure 5, (Section 7). In the event that the carrier's representative is unwilling to cooperate in this fashion, the SC will identify, in the proper position on the Chain-of-Custody document, the shipment number, the date of receipt, and sign off, attaching a copy of the shipping log for that particular container.

#### 8.5 EXAMINATION OF CONTAINER CONTENTS

Unless the shipping container contents are marked "hazardous" the SC will proceed to open the sample container. If the SC had not previously identified the project sheet appropriate for these samples, the SC will attempt to ascertain immediately the origin of the samples found in this container and obtain the appropriate project sheet. If a project sheet is not found, the SC will lock up the samples and notify the lab manager as described in Section 2.0. The SC will identify whether or not all the samples have arrived intact, whether or not the labels are intact and attached properly, and whether or not the samples have leaked in any fashion. The SC will also identify any shipping instructions, field instructions, or any other materials that may be present in the shipping container.

#### 8.5.1 Chain-of-Custody Shipments

Should the SC identify the shipping container as a Chain-of-Custody project, the SC will immediately follow the procedure outlined in Section 4.0, "Chain-of-Custody Samples".

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#### **8.6 PROJECT VERIFICATION**

The sample coordinator, having opened the shipping container and examined all the samples, will verify that the project sheet matches the samples, the number of samples received is consistent with the project sheet, and that the requirements identified on the project sheet are consistent with any paperwork obtained which will include the project sheet and any other documents in the sample container. The project files will be kept by the SC in a locked filing cabinet. If all required project information is not complete, the SC will fill out a Problem Project Sheet (see Section 5.2) and turn it over to the Project Manager.

#### 8.7 LABILE SAMPLE DISTRIBUTION

Should the SC identify labile samples within the shipping container, (i.e. coliforms or nitrites) for which there is a very short holding time and a need to rapidly move the samples into the laboratory, the SC will make every effort to immediately log-in those samples. Should log-in be delayed, the SC will coordinate with the responsible analytical group in order to move the samples into analysis. The coordinated effort will included means by which the SC can label the samples after log-in and insure that the results correlate with the proper samples. The SC will provide computer generated sample identification to the responsible analytical group. It will be the responsibility of the SC, once labile samples have been distributed to the laboratory to insure that those samples are properly logged in and that they are labeled with properly sequenced numbers. The agreement that is made between the SC and the appropriate laboratory manage or laboratory supervisor will be based on the premise that the SC understands that he/she is ultimately responsible and will be held accountable for any samples that are lost in such a movement. Consequently, the SC will find the samples that are labile and apply the necessary labels.

If a shipping container is labeled "Hazardous", the SC will immediately notify the laboratory supervisor who will determine the extent of hazard and/or the manner in which the samples will be handled. The supervisor will involve the laboratory manager as needed in resolving questions of hazardous samples.

#### FIGURE 11

#### POSITION DESCRIPTION FOR SAMPLE COORDINATOR

#### GENERAL

The Sample Coordinator (SC) is responsible for the receipt, log-in, and storage of all client samples at WWES. The SC is responsible for the receipt, storage and custody of all Chain-of-Custody (COC) samples including distribution of COC samples to lab personnel per WWES COC procedures (section 4.0, WWES Log-in Procedure). In order to ensure the successful analyses of samples, it is critical that the SC obtain and communicate to Project Manager, lab supervisors, and lab personnel, all information necessary for the processing interpretation and reporting of all samples analyzed.

#### **QUALIFICATIONS**

High School Diploma and a minimum of 2 years of college or equivalent. A knowledge of chemistry and testing procedures helpful. Excellent verbal, written and organization skills, including a propensity for detail necessary for successful completion of job.

#### REPORTING RELATIONSHIPS

The SC will report to the laboratory manager. The SC will communicate closely with the Director and Project Managers to obtain project information.

#### 8.8 CHAIN-OF-CUSTODY SAMPLES

# 8.8.1 Continuance of Log-In Procedures for Chain-of-Custody Samples

All samples in the possession of WWES under Chain-of-Custody (COC) procedures must be traceable from the time the samples are received at the WWES door (or collected by WWES staff) until results are reported and sample disposition has been determined from the client. For any samples that may be collected during enforcement investigations, under litigatory requirements, or evidentiary in nature, Chain-of-Custody procedures are required.

#### 8.8.2 Examination of Container Contents

Although Section 8.4.2 under Sample Receipt discusses the thorough examination of container contents, the proper examination of a container which is involved in a Chain-of-Custody procedure is even more important. For example, should the sample labels be mismarked or a particular sample to somewhat strange in nature, it is necessary to note all observations and deviations from the project sheet. It is better to be overly observant than to allow possible anomalies to go unnoticed. It is the SC's responsibility to examine whether or not each of the sample containers are individually sealed, whether those seals are intact, whether a sampler's initials are on the seals, and whether or not the paperwork matches the contents of the package. In addition, the SC must note whether or not all the dates and times are consistent, and whether or not the sample description on the paper work matches the description on the sample container.

# 8.9 PROJECT VERIFICATION

In the same manner in which the examination of the container contents is critical to a COC project, the verification of the project is equally important. These project verification steps include not only the need to follow the requirements identified in Section 8.6, but also thorough examination of all aspects of the project and the consistency of all the paper work involved with those particular samples in that shipping container. It is also important that the SC place in the COC project file: the shipping document; a signed Chain-of-Custody document including the sign off from the shipper's representative (See Section 8.4.3); a copy of the project sheet; a copy of the Project Approval Form is appropriate; a copy of the filed sampling report if appropriate; and originals of all paperwork received for the project. The COC project file is kept in locked storage in the possession of the SC.

#### 8.10 CHAIN-OF-CUSTODY LOG-IN

The log-in procedure identified in section 8.15 titled "Log-in", is followed in the same manner for Chain-of-Custody samples with a few modifications. Those areas which are changed are addressed in the following sections:

- Sample Storage
- Project Files
- Laboratory Access
- Data Storage

#### 8.11 CHAIN-OF-CUSTODY SAMPLE STORAGE

All samples received under Chain-of-Custody procedures will be kept under locked storage and will be distributed for analysis to the laboratory only when the analyst has signed for the samples on the form shown in Figure 6, (Section 7). The SC or a designated representative will provide access to COC storage. Records of movement of all COC samples within the lab facility must be recorded.

#### 8.12 CHAIN-OF-CUSTODY PROJECT FILES

All Chain-of-Custody project files will be kept in a project folder in a locked cabinet with all related documents and paperwork relative to those files.

#### 8.13 MAINTENANCE OF LAB CUSTODY

Laboratory custody must be consistent with all the Chain-of-Custody requirements from the beginning of sampling to the final report. To this end, every analyst requiring access to the Chain-of-Custody samples will go to the SC for access to the COC locked sample storage. The SC will insure that the analyst signs for the receipt of all COC samples on the form shown in Figure 6, (Section 7) and that the analyst returns and signs in those same samples on the same day for which they were signed out. This documentation, after the completion of all analyses, will be placed in the locked Chain-of-Custody project file by the SC.

#### 8.13.1 Sample Custodian

The COC sample custodian at WWES will be designated as the Sample Coordinator (SC). The SC is responsible for following the COC requirements outlined in these procedures for all samples received at WWES.

#### 8.13.2 Lab Custodial Responsibilities

It will be the responsibility of every analyst signing for a Chain-of-Custody sample or samples to insure that; a) these samples are kept in a minimum access

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facility; b) they are within their possession during the particular period during which they are being analyzed; and c) the analyst returns those samples to the Chain-of-Custody lockup in the manner prescribed. The analyst will sign out and return the samples to COC lock-up on the same day. The analyst will be using the SC as the sample custodian for all COC samples. Due to the legal implications for the client of breaking the COC procedures and possibility of legal action that could be taken against WWES, errors in the execution of Chain-of-Custody procedures will not be tolerated.

#### 8.14 CHAIN-OF-CUSTODY SAMPLE DISPOSAL

All samples received for COC procedures will be stored in the WWES COC lock-up facilities until a final report is issued. It will be the responsibility of the Project Manager, in cooperation with the SC, to obtain information from the client relative to the length of time the COC samples will be stored. It is anticipated that for long term storage, i.e. more than 30 days, the client will reimburse WWES 'n appropriate rate for keeping completed samples under Chain-of-Custody procedures. No Chain-of-Custody samples may be discarded until written permission is received from the client relative to disposal of those samples.

#### **8.15 LOG-IN**

#### 8.15.1 Introduction

After the Sc has inspected the shipping containers, the project sheets, the samples and any documentation required in Sections 8.4 and 8.8, the SC will insure that all pertinent information is entered on the project sheet. There are specific areas of the project sheet that are to be completed by the SC, i.e., date and time received. The WWES project sheet is included as Figure 2, (Section 7).

Minimum information required for log-in include:

- Client's name and Client contact, as well as client #, is assigned.
- The due date
- The analytical test or test codes or group tests
- Specific project comments
- Contract requirements
- Contract number
- Pricing if necessary
- The approval for non-routine projects
- Chains-of-Custody, if required
- Specific report requirements

# 8.15.2 Project Problems

If any of the information identified in sub-section 8.15.1 is missing, the SC will immediately notify the Project Manager, via a Problem Project Sheet, Figure 4, (Section 7) of the discrepancy. The Project Manager will make all reasonable efforts to insure that the answers are provided to the SC immediately.

Simple Project Sheet deficiencies such as client number, extra comments, or the contract number, should not prevent log-in. The SC will proceed with log-in addressing the unknowns as subjects that must be changed or modified once the information is received. It is the responsibility of the SC to log-in all samples as received at WWES whenever possible.

### 8.15.3 Samples on Hold

When there is a considerable amount of inadequate information on a project sheet, i.e. a missing test, or broken samples, the entire project will be placed on hold until the information is available or the corrective actions have been taken to insure that NSF is not held responsible for a poorly handled project. The SC will notify the Project Manager via a Problem Project Sheet as to the hold status of the project and the reasons for the hold. The Project Manager will make every attempt to quickly identify the necessary actions that will be taken for those samples or the remaining samples for that project. The Project Manager may approve log-in of the remaining samples for a portion of the project in order to insure that the project progresses. Projects that are placed on hold will be locked in a "project hold" area, (like the Chain-of-Custody sample storage area) so that those samples are not lost or confused within the system. The SC will insure that those samples are retrieved and logged in as soon as the appropriate changes have been made and the samples are freed for log-in.

#### 8.15.4 Handling Labile Samples

All samples received by the SC that are labile in nature, i.e. coliforms, need to be logged into the facility in a very rapid fashion in order that they may e attended to within the analytical holding time. The most labile of all samples are the microbiological samples, which must be forwarded to the micro lab as soon as possible. The SC and the Project Managers responsible for micro work will attempt to insure that appropriate information is available to the SC in order that the SC can assign numbers for all labile samples. These numbers can be assigned in advance and samples may be logged into the system as soon as they are received. Samples such as nitrites, which are labile but have a somewhat longer holding time, will usually be logged into the system like normal samples. However, slow shipment or other problems may require the lab to initiate the analyses immediately. In such a case, assuming a project sheet was initiated in

advance o sample receipt, the SC can assign laboratory in an expedient fashion. The SC will make all efforts to insure that samples move through the laboratory in a timely fashion when holding times are of utmost importance to the proper completion of the analytical requirements.

#### 8.16 COMPUTER LOG-IN

It is anticipated that all samples received at WWES will be logged on to the computer by the SC. The computer assigns a sequential number to every sample. Additional codes such as the month and the year of the samples may be added in front of the sequential number for continuous identification of these samples. The SC will have the computer generate these sequential numbers for each sample in every project. A project identifier will be printed on the labels which are attached to every sample and every aliquot of a sample.

#### 8.17 SAMPLE SPLITTING FOR THE CHEMICAL LABORATORY

The WWES Project Manager will attempt to insure that all samples received at the WWES facility are received in the appropriate containers with the correct preservatives (Samples which must be split at log-in are subject to added error). The labels and the appropriate preservatives are depicted in Figure 12.

## 8.17.1 Bottles and Preservative Requirements

The WWES analytical facility has a series of bottle and preservative requirements that must be met before the log-in of samples into the laboratory. In the event that WWES is unable to provide sample bottles, or circumstances prevent the splitting of samples in the field, the SC will provide sample splitting services. These services will include taking the sample as received and subsampling it into the appropriate bottle and preservative requirements as set forward on the attached list of bottle and preservative requirements.

#### 8.17.2 Inorganic Samples

The SC will insure that sufficient sample volume is available before initiating the splitting of a sample. If uncertain, the SC will involve the laboratory supervisors in order to insure that all areas of the lab have sufficient samples. In the event that sufficient samples does not exist, the SC will identify the sample as a problem and will notify the Project Manager immediately for resolution. The sample will be logged in only after a resolution has been reached.

#### 8.17.3 Organic Analysis

When a bulk sample arrives for organic/inorganic analysis and sufficient sample exists, the SC will transfer the sample to the organic preparation supervisor who

# Environmental Laboratory Division

# WW Engineering & Science 5555 Glenwood Hills Parkway, SE Grand Rapids, MI 40588 • (616) 942-9600

| Date Requested:   | / | / | Date Due: | / | 7 |          |
|-------------------|---|---|-----------|---|---|----------|
| Dispatched By:    |   |   |           |   |   | <u> </u> |
| Project:          |   |   |           |   |   |          |
| Project Manager:_ |   |   |           |   |   |          |
| Project No:       |   |   | •         |   |   |          |
| Location:         |   |   |           |   |   |          |
|                   |   |   |           |   |   |          |

Sample Inventory and Master Bottle Packing List

| Sample                                | Sample      | L       |          | San | npk          | e S  | ub-          | <u>Po</u> | <u>rtic</u> | enc | <u>-P</u> 1 | <b>'es</b> | erv      | <u>ativ</u> | <b>10</b> 8 | ınd     | Ta       | gg       | ing      | C        | ode      | :3                           |  |                |          |
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Indicate Sample Sub Portion with an X

Multiple Sub Portions for the same Bottle Type can be identified by Entering the Number Needed

| NO. | DESCRIPTION                                | PRESERVATIVE  | TAG COLOR FILTERED |
|-----|--|---|--------------------|
|     | Waters                                     |   |                    |
| 1   | 40 ml Vial for Purgeable Organics          | 1+1 HCL Yes / No<br>Cool to 4° C                        | Yellow             |
| 2   | 1000 ml Amber Glass Non Purgeable Organics | Cool to 4° C  | Salmon             |
| 3   | ml Plastic - Non Preserved .               | Cool to 4° C  | Green              |
| 4   | ml Plastic - Nutrients                     | pH < 2.0 w/H <sub>2</sub> SO <sub>4</sub>               | Blue               |
| 5   | mi Amber Plastic - Cyanides                | pH to > 12 w/NaOH                                       | Light Blue         |
| 6   | mi Plastic - Metais                        | pH to <2 w/HNO <sub>3</sub>                             | Red                |
| 7   | 1000 ml Glass - Oil & Grease / TPH         | pH to <2 w/H <sub>2</sub> SO <sub>4</sub>               | Dark Blue          |
| 8   | 125 ml Whirl Pac Bag / Bottle Bacteria     | Cool to 4° C  | Brown              |
| 9   | 500 ml Glass - Sulfide                     | 0.5 ml Zing Agetate<br>+ 0.5 ml NaOH to pH >0           | Light Green        |
| 10  | 250 ml Amber Glass - TOX                   | pH to < 2 w/H <sub>2</sub> SO <sub>4</sub> Cool to 4° C | Lilac              |
| 11  | 40 ml Amber Glass - TOC                    | pH to < 2 w/H ,SO 4<br>Cool to 4" C ·                   | Pink               |
| 12  | 2000 ml Plastic - Radiological             | pH to < 2 w/HNO <sub>3</sub>                            | Gray               |
| 13  | 500 ml Amber Glass - Phenois               | pH to < 2 w/H <sub>2</sub> SQ                           | Brown ··           |
| 14  | 250 ml Amber Glass - Formaldehyde          | Cool to 4° C  | Orange             |
|     | Solls                                      |   |                    |
| 15  | ml Wide Mouth Plastic                      | Cool to 4° C  | White              |
| 16  | ml Wide Mouth Amber Glass                  | Cool to 4° C  | Manilla            |
| 17  | 125 ml Vial for Purgeable Organics in Soil | Cool to 4° C  | Light Yellow       |
| 18  | Other                                      |   |                    |

will split the organic aliquots and return all aliquots to the SC. The remaining sample will then be returned to the SC who will split off the inorganic aliquots into the proper preserved containers.

#### 8.17.4 Solid Samples Splitting

When solid samples, such as sediment or soil, are to be received at WWES, every attempt will be made by the Project Manager and field sampling personnel to insure that two samples are provided as replicates for the appropriate tests. One of these samples will be assigned to the organic facility; the other will be assigned to the inorganics facility. If only one sample is received and if organic analyses are required, the organics preparation chemist will be responsible for the initial splitting of the sample. Solid samples will be made homogeneous by either one or all of the following manners:

- Stirring especially when volatile organic analytes are required
- · Air Drying and Grinding
- Particle separation (Sieving)
- Quartering by ASTM Procedures

The lead organic chemist and the SC are responsible for the decisions on how a solid sample will be split. Problems or concerns which may arise on a solid sample will be addressed to the Project Manager and the laboratory manager for resolution. After the organic portions have been removed or split, the remaining sample will be provided to the inorganic facilities for any further splitting they deem necessary.

#### 8.18 SAMPLE LABELING

All samples received at the WWES facility will be labeled by the SC at the time of login. These labels will include information such as the requested sample number, the client number if supplied, the contract, the WWES project number, and/or the client. It is anticipated that sequential sample labels will be provided by the computer after the SC has logged the project into the computer.

#### 8.19 DISTRIBUTION AND STORAGE

Logged samples will be taken by the SC to the appropriate walk-in cooler for cold storage or to the room temperature storage area indicated for metals.

COC samples are stored as set forth in Section 4.0.

#### 8.20 PROJECT FILES

# 8.20.1 Routine Project Files

The SC will obtain a manila folder and label that manila folder with the name and number of the project. The folder will indicate the WWES project number, the WWES contract number, and Chain-of-custody if applicable. With the agreement of the laboratory supervisor (lead), the project manager, and the laboratory manager, a particular project folder may include a series of projects logged in under sequential numbers. An example would be a daily log-in for the same project for a week or month before a new project folder is generated. It is, however, the responsibility of the SC to insure that all logged projects are filled in a project file folder.

# 8.20.2 Chain-of-Custody File Folder

The SC, upon logging in any Chain-of-Custody project, will provide the same type of manila folder project file, as discussed in Section 5.7.1, for each project. However, the project folder will be maintained in the locked Chain-of-Custody file and cabinet and will be kept by the sample coordinator.

#### 8.21 SAMPLE STORAGE

# 8.21.1 Non Chain-of-Custody Storage

The SC, after completing all the log-in processes of various samples connected with a particular project, will store the samples in the designated areas in the WWES laboratory.

- Routine Water and Solid Samples: Samples which need to be refrigerated will be stored in the walk in facility designated for all routine water and soil samples.
- Routine Volatile Water and Solid Samples: All these samples will be placed in the designated VOA refrigerator(s) located within the analytical facility. No other samples or standards may be stored in the VOA refrigerator(s).
- Routine Water and Solid Samples for Metal Parameters: The preserved water samples and solid samples, which are not preserved, may be stored on shelves designated for the metals analysis.
- Odoriferous and Hazardous Samples: These samples will be stored in a hooded facility within the laboratory which is designated for Odoriferous and hazardous samples. These samples will be identified to the lab

personnel and noted on the log-in procedures in order to insure that the lab personnel are aware of the problems with these samples.

#### 8.22 CHAIN-OF-CUSTODY SAMPLE STORAGE

All samples that are involved as physical evidence in a legal procedure or simply identified as Chain-of-Custody will be handled under certain procedural safeguards. These safeguards have been tentatively identified in section 4.0 but for purposes or reiteration are again addressed below:

NOTE: For any legal proceedings, the court must be shown that the laboratory is a secured area, that all samples have been stored in a secured fashion, and samples can be accounted for at all times.

#### 8.22.1 Chain-of-Custody Water and Solid Samples

All samples of this nature will be stored within the locked confines of the Analytical Laboratory. Access is only available to authorized personnel.

#### 8.22.2 Water and Soil Samples for Metals

#### 8.23 GENERAL LAB SECURITY

Access to the WWES lab will be handled in a secured fashion restricting entrance only to those people designated as having access to the laboratory facilities. Restricted access applies to all areas in which samples are stored or analysis takes place. It will be the responsibility of all the analysts, as well as the supervisors and the SC, to insure that the safeguards employed, including locked doors and limited access, are followed and maintained at all times.

# 9.0 DATA HANDLING, REPORTING, RECORDKEEPING AND VALIDATION

#### 9.0 DATA HANDLING, REPORTING, RECORDKEEPING AND VALIDATION

There are two significant aspects of any analytical procedure:

- The selection and use of a method appropriate for the analyte and matrix
- The collection, control and interpretation of the data generated.

Encompassing these two components is the Quality Assurance program. The QA program provides means by which method selection can be validated, the method can be controlled and the appropriate data generated, displayed and reduced.

The following sections deal with error, data handling, data validation, data reporting and data recordkeeping.

#### 9.1 ERROR: IT'S NATURE AND SIMPLE STATISTICAL CONCEPTS

#### 9.1.1 Random Errors

Repeated analysis of identical aliquots of a homogeneous sample does not give a series of equivalent results. The results will differ among themselves and they will be more or less scattered about some value. The scatter can be attributed to random error, so named because the prediction of the sign or magnitude of the error of any particular result is not possible at the time of analysis.

One therefore, says that each result must have an uncertainty attached to it, and can be regarded only as an estimate of the true value. Generally that estimate will differ from the true value. Random errors are caused by uncontrolled and/or uncontrollable random variations in factors which affect analytical results, i.e. variations in the volumes of the reagents added, variations in the concentrations of reagents, variations in the time allotted for the chemical analysis, a contaminated glassware, poor quality reagents, instrumental fluctuations. Among the various texts that are available discussing errors, the terms repeatability, reproducibility and precision have been used to denote the scatter of results. The term "precision" will be used throughout this manual and is the most common term used for random error in this country and especially by the EPA.

Precision does improve as the scatter among results becomes smaller. All analytical results have random error present which necessitates statistical techniques to evaluate the results and to provide correct inferences of the true value of the result.

#### 9.2 SYSTEMATIC ERRORS

Systematic errors are indicated by the tendency of results to be greater or smaller than, the true value. It is necessary to take care in exactly defining systematic error because

the analysis is also subject to random error. The mean of n analytical results on the same sample approaches a definite value u as the number of results increases indefinitely. When u differs from the true value Tau results are said to be subject to systematic error of the magnitude B, wherein B is equal u minus Tau. Bias is the term used synonymously with systematic error and will be used in that fashion throughout this manual. Analytical methods, which are subject to interferences from substances present in the sample, or methods that only recover a fraction of the material present are an example of systematic error.

It is impractical to make an indefinitely large number of analysis on a single sample in order to determine the true value of u is known. At the same time a practically obtained value for a sample that is based on minimal analysis is subject to random error, so that the experimental estimates of bias will also be subject to random error. Therefore, statistical techniques are also required when bias is to be estimated.

The basic difference between random and systematic error is that, in principal, the latter may be predicted so that a correction can be made to eliminate its effect. An example of this allowance can be accounted for in the effect of fluoride in the determination of aluminum by absorbance measurements. This effect is overcome by adding to the calibration standards an amount of fluoride equal to the fluoride content of the sample. The added fluoride in the calibration standards then eliminates the systematic error of fluoride interference. However, it must be recognized that the complete elimination of systematic error may require such detailed knowledge of the properties of the sample that the correction of the analytical system is impractical and would in fact increase the amount of random error. Thus, in all applications where unbiased results are necessary, the approach to be used is to devise and use analytical systems capable of giving results which have negligible systematic error.

#### 9.3 TOTAL ERROR

Some analysts use the term accuracy to denote only systematic error. The term accuracy as applied in this manual will denote total error of the results. In other words, accuracy represents the combined systematic and random error of the results and, therefore, the accuracy of an analysis improves as the total error becomes smaller. For the purposes of visually seeing random and systematic error, Figure 6-1 should be referred to for any easy identification of the various types of error.

#### 9.4 STATISTICAL TECHNIQUES

Statistical techniques are essential to the measurement of analytical error. This manual and this section recognize that many analysts have had little experience with statistical technique. This section is, therefore, written in such a way as to explain simple but basic concepts of the statistical approach and to describe the particular techniques most commonly required in dealing with analytical errors. There are a large number of text

books dealing with statistics and this particular section does not attempt to replace these books. The intention is merely to present the essential aspects in the simplest manner possible. Certain approximations have been used when considered appropriate and no previous knowledge of statistics has been assumed. Should the analyst be interested in consulting additional texts for a more rigorous and detailed treatment of the subject, he is referred to the references at the end of section 9.0.

Analysts who are unfamiliar with statistical approach, may find this section on first glance rather complicated. In order to understand statistics for the QC function, it is important not to be put off by the first impression.

The fundamental statistical concepts are essentially simple and equivalent to the intuitive common sense, or perhaps scientific approach, adopted by any good analyst.

#### 9.4.1 Random Error Distribution

If the results from the analysis of numerous aliquots of a homogeneous sample are plotted on a histogram, it is generally found that the proportion of the results deviating from the mean increased, i.e., as the deviation of the results from the mean grows broader. In other words, the probability of obtaining a random error of a given size decreases as the size of the error increases. The basis of statistical techniques is to quantitatively estimate the probabilities of errors of different sizes so that one can deduce the probable random error of a particular analytical result. If the analyst were to increase the number of analysis of a single sample indefinitely, and the size of the intervals used for plotting the histogram were decreased, the latter would tend to smooth the curve. This limiting curve is the frequency distribution of results and defines a relationship between the magnitude of the result and the probability of obtaining such a value. Throughout this manual, it will be assumed that the analytical results follow the normal distribution which is defined by the following equation:

p(x) =

Where:

- = the mean of all the conceptionally infinite number of results.
- = the standard deviation of results
- p(x) = the probability density which is interpreted by noting that the probability of obtaining a result between the values a & b is the area of the curve between those values.

and this interval can be evaluated given the equation for P(X).

The peak of this distribution curve occurs at x=u, the theoretically perfect mean established by an infinite number of results. The width (which is indicated by the

scatter results) is determined solely by the standard deviation of the test. For example, 95% of the area under the curve, i.e. 95% of all results, is enclosed within the limits plus or minus 1.96. Such properties allow limits for the uncertainty of an individual analytical result to be calculated. Taking the current discussion, for example, on no more than 5 occasions in one hundred will the result differ from the mean u be more than 1.96. Thus, an analyst may attach to a result limits that define the range in which the true mean is expected to lie. The statement, R-1.96 is less than u which is less the R+1.96, is an accurate statement on 95% of all occasions. "R" in this particular case would stand for the result. By referring to texts on statistics, there are statistical tables which included a tabulation of areas enclosed between specific limits as an analyst might want to define them. It should be noted that the distribution is always symmetrical about the mean. In other words, if one is using the 1.96 levels 5% of the results will be outside of the range of u +/- 1.96, but only 2.5% of all results will exceed u + 1.96 and 2.5% of the results will be less than u - 1.96.

Focusing this into a discussion more pertinent to the laboratory and, perhaps more viable with respect to occurrences within the laboratory, let us discuss the rare exception in which an analyst is taking 20 tests on a particular sample using the 1.96 level. Considering that 5% of the results will lie outside that level, the analyst has 1 chance in 20 of missing the true value outside the stated confidence range. At the same time one can decrease this chance by increasing the allowable range. For instance, if the range is R =/- 2.58 the results will be included on 99% of the occasions or 99% of the tests. However, by increasing the confidence limit, one is also increasing the uncertainty in the true value. In this case, uncertainty can be decreased by taking the mean of several analytical results or by decreasing the value.

These statistical concepts allow valuable quantification of the random error of an analytical result and emphasize that decisions, based on the significance of the result, have some risk of being wrong. Knowledge of the standard deviation, of the results is, therefore, vital in reaching objective decisions. Use of the standard deviation will be explained in the following sections dealing with data handling and validation.

#### 9.4.2 Data Handling, Reporting, Recordkeeping

A flow diagram, Figure 1, delineates the original and procedural steps in data generation.

The initiation of an analysis starts with the completion of a project approval form. The information is computer entered. The computer entry internally creates a report form and inventories the analysis by parameter or compound. The computer entry function of all analytical work requests is a shared responsibility

of the sample coordinator and data coordinator. A copy of the analysis request form is manually inserted into a three ring binder notebook for laboratory reference use. The maintenance of the laboratory job reference notebook is a responsibility of the sample coordinator. The group leader/supervisors requests from the data coordinator (D.C.), the computer generated analytical bench sheets for a given parameter each morning or the prior day. The samples and parameters testing sequence is dictated by a weekly work schedule. The weekly work schedule is developed manually each week by the group leaders/area supervisors and approved each week by the laboratory manager. The schedule is developed from a computer printout that inventories and ages by project job or parameter. Contractual due dates and sample holding times are the compliance criteria by which all schedules are judged.

The bench sheets examples are shown in Figure 7, 8, 9. The bench sheets identify to an analyst the proper samples to analyze that day. The analyst lab notebook and the bench sheets constitute the two raw data reporting locations. The content of the laboratory notebook is defined in an earlier section, 7.3.7. The analyst completes the benchsheet information, attaches a drawn calibration curve and follows the analytical sample sequence identified in section 10.0. The analyst identifies which sample(s) were utilized for precision and accuracy determinations. The analyst will assess the data set as being in control or not. The assessment will be described in the data validation section to follow. The analyst will submit to respective group leaders or supervisors all of the abovementioned data and a written statement that the data set is in control for their review. An approved data set is signed off and the group leaders/supervisors transfer the approved data to all appropriate worksheets in the laboratory job reference notebook. The bench sheets and calibration curves are permanently stored. The last entry into the worksheet constitutes a completed project subject to computer generation of a preliminary report. The group leader/supervisors provide the DC with the approved worksheets for computer entry and preliminary report generation. The remaining activities related to preliminary report, final report generation and review and project filing are identified in this manual under sections 7.3.14, 7.3.15 and 7.3.16 respectively.

#### 9.5 DATA VALIDATION

The data validation process includes a set of computerized and manual checks at various appropriate levels of the measurement process.

The data validation process starts with the laboratory analyst. The analyst verify in their lab notebook that all method specific operational parameters are utilized or met. This information is specifically documented in all instrument logbooks. The analyst then verifies that the calibration of the equipment is linear and documents this in the instrument logbooks. If the operating parameters of a particular method are modified, it

should be written in the analyst lab notebook and approved via signature by the group leader/supervisor in the lab notebook. A non-calibrated system must be identified by the analyst and corrections made to achieve calibration prior to sample analysis.

The generation of sample data by an analyst will include the generation of quality control data for each sample set. The monitoring of method blanks, sample spikes, method spikes and sample duplicate analysis is accomplished by the utilization of Schwart Quality Control Charts. All quality control data is entered on the precision and accuracy data summary form, Figure 11a. The analyst computes the data precision and accuracy and compares the computed value to the acceptance intervals identifies on the form for that parameter, method, and matrix. The computed value will be determined in control if it lies within the acceptance interval. If the computed value is deemed out-of-control the data set is not submitted for supervisor approval but is brought immediately to the attention of the supervisor and quality assurance officer that an out-of-control condition exists. Jointly, a review is conducted to determine the cause(s) and conduct corrective action. The data set is rerun once the corrective actions have taken place and the new data reviewed as stated above.

The DC receives all the completed precision and accuracy data summary forms and enters the data into the laboratory quality control computer system. The system produces summary reports each day of all quality control data generated for review by the quality assurance officer. The computer system also generates all Schwart Control Charts for method blanks, method spikes, sample duplicates and sample spikes. The charts are permanently maintained and reviewed each week by the group leader/supervisor and the quality assurance officer. The weekly generated charts provide an accurate review of all recently (last 30) qc data points and allows the monitoring of data trends or other anomalies to the system.

# 10.0 GENERAL QUALITY CONTROL PRACTICES

#### 10.0 GENERAL QUALITY CONTROL PRACTICES

The Quality Assurance/Quality Control practices at WWES are based on several of the following government guidelines:

- "Handbook for Analytical Quality Control in Water and Wastewater Laboratories "EPA 600/4-79-019, March 3, 1979
- The Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act 40 CFR; July, 1990.
- Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples" EPA 600/8-80-038 June 1980.
- ASTM
- Test methods for evaluating a solid waste; USEPA SW-846; Third Edition, Revision 0.
- 10.1 The quality control types normally analyzed during sample analysis includes the following: Initial Calibration Blank (ICB), Initial Calibration Verification (ICV), Method Preparation Blank (MPB), Laboratory Control Sample (LCS), Sample Matrix Spike Duplicate (MSD), Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB).
- 10.2 The frequency of which these QC types are performed during the analytical run is usually stated within the analytical method. The general frequency over-all of these types, and their respective order within the analytical run is as follows: (following instrument calibration).

| Туре                                | -Frequency  |
|-------------------------------------|-------------|
| Initial Calibration Blank           | 1-per batch |
| Initial Calibration Standard        | 1-per batch |
| Sample #1                           | ***         |
| Sample #2                           |             |
| Sample #10                          |             |
| Method Preparation Blank            | 1-per batch |
| Laboratory Control Sample           | 1-per batch |
| Sample Matrix Spike                 | 10%         |
| Sample Matrix Duplicate             | 10%         |
| Continuing Calibration Blank        | 10%         |
| Continuing Calibration Verification | 10%         |

Any high level concentrations of analyte will be followed by a blank.

- 10.3 The level of internal laboratory quality assurance effort for the following is divided into 4 different categories:
  - 1. Routine Analytical Services (RAS). No special reporting requirements are required.
  - 2. Reportable Analytical Services (REP). For this type, batch quality control is reported for all analytes.
  - 3. Special Analytical Services (SAS). Each matrix type for a particular submittal will have internal QC performed on these particular samples at the appropriate method frequency.
  - 4. Quality Assurance Project Plan (QAPP). This level of QC encompasses all aspects of the SAS type with full data deliverables similar to CLP reporting packages.
- 10.4 The fundamental QA objective with impacting accuracy, precision and sensitivity of laboratory analytical data is to achieve the QC acceptance criteria established for each analytical method and matrix type.

The control limits established for each method are based on  $\pm$  3 standard deviations from the analytical mean. Also encompassed are method advisory limits if provided within the analytical methodologies.

The standard operating procedures that would lead to an outlier being identified and the resulting corrective actions is described in section 9.0, Data Reporting, Validation and Handling. In general, if an out-of-control result occurs the analyst will identify it as such and report the occurrence to the Group Leader and/or Area Supervisor. The Group Leader and/or Area Supervisor will review the data with the analyst to identify the problem, implement a corrective action(s) and then re-analyze the sample(s). The Group Leader and/or Area Supervisor will report the out-of-control occurrence to the Quality Assurance Manager that day in writing (Figure 13). The corrective action(s) will be identified in the analyst notebook and in writing to the QA Manager.

9/91

# FIGURE 13

# **Analytical Quality Control Occurrence Report**

| Parameter:                            |                                       |             | ·           |     | •            |             |             |
|---------------------------------------|---------------------------------------|-------------|-------------|-----|--------------|-------------|-------------|
| Method:                               |                                       |             |             | •   |              |             |             |
| Date:                                 |                                       |             |             |     |              |             | . ;         |
| Analyst:                              |                                       | · ·         | _           |     | •            |             |             |
| <b>Description of Occurrence:</b>     |                                       |             |             | ,   |              |             |             |
|                                       |                                       |             |             |     | : .          |             |             |
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| Analysis of Occurrence:               | ÷.                                    |             |             |     | ·            |             |             |
|                                       |                                       |             |             |     |              |             |             |
|                                       |                                       |             |             |     | · · · · · ·  |             | ·           |
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| Disposition of Data:                  | . 1                                   |             |             |     |              |             |             |
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# Appendix F

WWES Standard Procedures for U.S. EPA Method 8021

#### PURGEABLE HALOCARBONS EPA METHOD #8021

#### 1.0 SCOPE & APPLICATION

This method covers the determination of 60 purgeable halocarbons and aromatics. This is a purge and trap gas chromatographic (GC) method applicable to the determination of the compounds listed in Table 1, and is used for the characterization of groundwater, soils, sludge, water-miscible liquid waste, and non-water-miscible waste of any or all of the listed compounds. A second analytical technique (dual column or GC/MS) can be utilized to confirm any measurements.

#### 2.0 METHOD SUMMARY

An inert gas is bubbled through a 5-ml water sample contained in a specially designed purging chamber at ambient temperature. The organics are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the organics are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the organics onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the organics which are then detected with a photoionization detector and a halide-specific detector (Tracor Hall-1000) in series.

The method provides an optional gas chromatographic column that may be helpful in resolving the compounds of interest from interferences that may occur.

#### 3.0 ANALYSIS RATE

Instrumental analysis including blanks, standards, methods, matrix spikes and duplicates will allow approximately 16 runs in an eight hour period. This is limited, however, by the sample matrix.

#### 4.0 INTERFERENCES

- 4.1 Impurities in the purge gas and organic compounds outgassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system should be avoided.
- 4.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A trip blank prepared from reagent water and carried through the sampling and handling protocol will serve as a check on such contamination.

4.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water in the same purging device to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high organohalide levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105°C oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

#### 5.0 MATERIALS & REAGENTS

#### 5.1 Chemicals

- Reagent water (organic free)
- Methanol (purge and trap grade)
- Pure stock standard materials
- Sodium Thiosulfate (ACS), granular or certified pre-mix standard

#### 5.2 Glassware and Hardware

- Various size volumetric flasks
- Various size microsyringes
- 15 ml screw-top vial with teflon-faced silicon septum
- 5/25 ml. gas tight syringes
- 40 ml. septum vial

#### 5.3 Instrumentation

- Tracor Models 540, 585 & 9000 Gas Chromatograph(s)
- Hall Electrolytic Conductivity Detector
- PE Nelson Turbochrome Data System
- Tekmar LSC-2; ALS 10 Place Automatic Purge & Trap Units
- Tekmar LSC-2; LSC-2000, ALS 1016, ALS 10 Place Automatic Purge and Trap Units

#### 6.0 SAMPLE COLLECTION, PRESERVATION, & HANDLING

All samples must be iced or refrigerated from the time of collection until analysis. If the sample containers free or combined chlorine, add sodium thiosulfate preservative (10 mg/40 ml is sufficient for up to 5 ppm Cl<sub>2</sub>) to the empty sample bottle just prior to

shipping to the sampling site. EPA Methods 330.4 and 330.5 may be used for measurements of residual chlorine. Field test kits are available for this purpose.

Grab samples must be collected in glass containers having a total volume of at least 25 ml. Fill the sample bottle just to overflowing in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. If preservative has been added, shake vigorously for one minute. Maintain the hermetic seal on the sample bottle until time of analysis.

All samples must be analyzed within 14 days of collection.

#### 7.0 STANDARD PREPARATION

#### 7.1. Stock Standard

Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol using assayed liquids.

Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store at 4<sup>o</sup>C and protect from light.

All standards must be replaced after one month, or sooner if comparison with check standards indicates a problem.

#### 7.1.1 Standard Preparation from Certified Solutions

Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source. (Supelco Purgeable A,B, and C prepackaged as 200 mg/L in methanol.)

Certified solutions are generally used as stock standards unless the analyst wishes to analyze a partial list, in which case standards are made volumetrically from neat compounds. An intermediate standard at 1 ppm is prepared in methanol by diluting the certified solution in a volumetric flask.

#### 7.1.2 Standard Preparation by Volumetric Method from Neat Compounds

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To make a 10,000 ppm stock standard, an amount of each analyte based on the density, and calculated with the formula below, is injected into a 100 ml volumetric flask partially filled with methanol. The flask is brought to volume and inverted three times (after replacing ground glass stopper) to insure proper mixing.

Example for Trichloroethylene:

$$\frac{1}{1.464} \times 1000 = 683 \text{ ul}$$

The stock standard is then diluted in methanol to obtain a 1 ppm intermediate standard.

#### 7.2 Working Standards

These standards are made right in the 5 ml sample syringe which contains 5 ml of organic-free water. These standards must be loaded into the purging device immediately to avoid volatilization.

#### **8.0 PROCEDURES**

#### 8.1 Sample Prep

- 8.1.1 Soils are extracted by placing 20.0 grams of soil sample into a 40 ml vial to which is added 10.0 ml of methanol. The vial is then fitted with a Teflon coated septa, shaken for 30 seconds, and refrigerated until analyzed.
- 8.1.2 Water samples require no preparation prior to analysis.

#### 8.2 Initial Calibration

A water blank is injected initially. If the blank shows a contamination peak, water must be purged and the blank rerun. If the water is free of analyte, the standards may be injected, beginning with the lowest and proceeding to the highest. A series of standards consisting of 1,2,5,10 and 100 ug/l concentrations, are normally employed in the generation of the calibration curve. Analyze each calibration standard according to section 8.3 and plot peak height responses vs. concentration of the standard.

Prepare a QC check sample containing 20 ug/l of each parameter. This check sample should be prepared from a source other than that used for the calibration standards.

Analyze the check sample according to section 8.3 for each parameter compare the percent recovery of the check sample to establish acceptance limits. If all parameters are acceptable, the analysis of samples can begin. If any of the recoveries are outside established limits, the analyst must repeat the test for the parameters that failed. If the second test fails, a new calibration curve must be created and the process repeated.

#### 8.3 Sample Analysis

Allow the sample to come to ambient temperature prior to introducing it to the syringe. Remove the plunger from a 5 ml syringe. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger. Vent any residual air while adjusting the sample volume to 5.0 ml. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe or place remaining sample in a 20 ml vial with no headspace at this time to protect against possible loss of Allow the sample to come to ambient temperature prior to introducing it to the syringe. Remove the plunger from a 5 ml syringe. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger. Vent any residual air while adjusting the sample volume to 5.0 ml. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe or place remaining sample in a 20 ml vial with no headspace at this time to protect against possible loss of analytes. Add 10.0 ul of the surrogate spiking solution, and inject sample into purging chamber.

Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

Generally, the retention time should vary no more than 0.1 min from the standard. If any questions, spike sample or confirm on another column.

If the response for a peak exceeds the working range of the system, prepare a dilution of the sample with reagent water from the aliquot in the second syringe and reanalyze.

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#### 8.4 Calculations

Four working standards (1,2,5,10, ppb) must be calibrated and plotted of peak height on the vertical axis versus concentration in ug/l on the horizontal axis.

Concentration of Purgeables = ug/l = (DF)(CC)

where: DF = any dilution factor

CC = value obtained from the calibration curve

#### 8.5 QC Requirements

#### 8.5.1 Method QC

For each analytical batch a laboratory control sample and method preparation blank must be analyzed and recoveries calculated and compared to established control limits.

#### 8.5.2 Matrix QC

A matrix spike and matrix spike duplicate must also be ran for every 20 samples of the same matrix (or once per batch of samples). A duplicate is usually ran on a positive sample. If there are no positive samples, a duplicate of the matrix spike should be run instead.

Spike samples with a shortened compound list, all compounds at 10 ug/l:

1,1-Dichloroethylene
Trichloroethylene

Chlorobenzene

Benzene

Toluene

#### 8.5.3 Continuing Calibration Verification

The calibration check standard or continuing calibration verification (CCV) standard must be analyzed at a frequency of 10%. The recoveries of the CCV must fall within an 85-115% recovery range.

#### 8.5.4 Surrogate

The analyst should monitor both the performance of the analytical system and the effectiveness of the method with each sample matrix by spiking each sample, standard, and reagent blanks with surrogate halocarbons. Prepare a stock spiking solution containing 10 ng/ul of 4-Chlorotoluene.

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Add 10 ul of this spiking solution directly into the 5 ml syringe for every sample and standard. Prepare a fresh spiking solution on a weekly basis. Recoveries of the surrogate compound must be monitored with acceptable windows established for each by calculating upper and lower control limits from the mean recovery. The control limits are based on  $\pm 3$  standard deviations from the mean.

#### 9.0 SAFETY

- 9.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potentially health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material handling data sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified for the information of the analyst.
- 9.2 The following parameters covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: carbon tetrachloride, chloroform, 1,4-Dichlorobenzene, and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

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#### 10.0 CORRECTIVE ACTIONS

See corrective Action SOP.

#### 11.0 REFERENCES

SW846, 3rd Edition, November, 1990.

TABLE 1
COMPOUND INFORMATION

| <b>*</b> • |       |
|------------|-------|
| Linear     | Kange |
| Linear     | Mange |

| Compound                    | (nnh)     | Formula  | CACNO    |
|-----------------------------|-----------|--|----------|
| Compound                    | (ppb)     | Formula C. W.                                    | CAS No.  |
| Benzene                     | 1.0-100.0 | C <sub>6</sub> H <sub>6</sub>                    | 71-43-2  |
| Bromobenzene                | 1.0-100.0 | C <sub>6</sub> H <sub>5</sub> Br                 | 108-86-1 |
| Bromochloromethane          | 1.0-100.0 | CH <sub>2</sub> BrCl                             | 74-97-5  |
| Bromodichloromethane        | 1.0-100.0 | CHBrCl <sub>2</sub>                              | 75-27-4  |
| Bromoform                   | 1.0-100.0 | CHBr <sub>3</sub>                                | 75-25-2  |
| Bromomethane                | 1.0-100.0 | CH <sub>3</sub> Br                               | 74-83-9  |
| n-Butylbenzene              | 1.0-100.0 | C <sub>10</sub> H <sub>14</sub>                  | 104-51-8 |
| sec-Butylbenzene            | 1.0-100.0 | $C_{10}H_{14}$                                   | 135-98-8 |
| tert-Butylbenzene           | 1.0-100.0 | $C_{10}H_{14}$                                   | 98-06-6  |
| Carbon tetrachloride        | 1.0-100.0 | CCl <sub>4</sub>                                 | 56-23-5  |
| Chlorobenzene               | 1.0-100.0 | C <sub>6</sub> H <sub>5</sub> Cl                 | 108-90-7 |
| Chloroethane                | 1.0-100.0 | C <sub>2</sub> H <sub>5</sub> Cl                 | 75-00-3  |
| Chloroform                  | 1.0-100.0 | CHCl <sub>3</sub>                                | 67-66-3  |
| Chloromethane               | 1.0-100.0 | CH <sub>3</sub> Cl                               | 74-87-3  |
| 2-Chlorotoluene             | 1.0-100.0 | C <sub>7</sub> H <sub>7</sub> Cl                 | 95-49-8  |
| 4-Chlorotoluene             | 1.0-100.0 | C <sub>7</sub> H <sub>7</sub> Cl                 | 106-43-4 |
| Dibromochloromethane        | 1.0-100.0 | CHBr <sub>2</sub> Cl                             | 124-48-1 |
| 1,2-Dibromo-3-chloropropane | 1.0-100.0 | C <sub>3</sub> H <sub>5</sub> Br <sub>2</sub> Cl | 96-12-8  |
| 1,2-Dibromomethane          | 1.0-100.0 | $C_2H_4Br_2$                                     | 106-93-4 |
| Dibromomethane              | 1.0-100.0 | $CH_2Br_2$                                       | 74-95-3  |
| 1,2-Dichlorobenzene         | 1.0-100.0 | $C_6H_4Cl_2$                                     | 95-50-1  |
| 1,3-Dichlorobenzene         | 1.0-100.0 | C <sub>6</sub> H <sub>4</sub> Cl <sub>2</sub>    | 541-73-1 |
| 1,4-Dichlorobenzene         | 1.0-100.0 | $C_6H_4Cl_2$                                     | 106-46-7 |
| Dichlorodifluoromethane     | 1.0-100.0 | CCl <sub>2</sub> F <sub>2</sub>                  | 75-71-8  |
| 1,1-Dichloroethane          | 1.0-100.0 | $C_2H_4Cl_2$                                     | 75-34-3  |
| 1,2-Dichloroethane          | 1.0-100.0 | $C_2H_4Cl_2$                                     | 107-06-2 |
| 1,1-Dichloroethene          | 1.0-100.0 | $C_2H_2Cl_2$                                     | 75-35-4  |
| cis-1,2-Dichloroethene      | 1.0-100.0 | $C_2H_2Cl_2$                                     | 156-59-4 |
| trans-1,2-Dichloroethene    | 1.0-100.0 | $C_2H_2Cl_2$                                     | 156-60-5 |

### TABLE 1 **COMPOUND INFORMATION**

(Cont.)

| Linear Range | · ·          |
|--------------|--------------|
| <u>(ppb)</u> | Formula      |
| 1.0-100.0    | $C_3H_6Cl_2$ |
| 1 0 100 0    | 0.17.01      |

|                           |              | /   | •          |
|---------------------------|--------------|---|------------|
| Compound                  | <u>(ppb)</u> | <u>Formula</u>                                | CAS No.    |
| 2,2-Dichloropropane       | 1.0-100.0    | $C_3H_6Cl_2$                                  | 590-20-7   |
| 1,1-Dichloropropene       | 1.0-100.0    | C <sub>3</sub> H <sub>4</sub> Cl <sub>2</sub> | 563-58-6   |
| cis-1,3-Dichloropropene   | 1.0-100.0    | C <sub>3</sub> H <sub>4</sub> Cl <sub>2</sub> | 10061-01-5 |
| trans-1,3-Dichloropropene | 1.0-100.0    | C <sub>3</sub> H <sub>4</sub> Cl <sub>2</sub> | 10061-02-6 |
| Ethylbenzene              | 1.0-100.0    | C <sub>8</sub> H <sub>10</sub>                | 100-41-4   |
| Hexachlorobutadiene       | 1.0-100.0    | C <sub>4</sub> Cl <sub>6</sub>                | 87-68-3    |
| Isopropylbenzene          | 1.0-100.0    | C <sub>9</sub> H <sub>12</sub>                | 98-82-8    |
| p-Isopropyltoluene        | 1.0-100.0    | $C_{10}H_{15}$                                | 99-87-6    |
| Methylene chloride        | 1.0-100.0    | CH <sub>2</sub> Cl <sub>2</sub>               | 75-09-2    |
| Naphthalene               | 1.0-100.0    | $C_{10}H_8$                                   | 91-20-3    |
| n-Propylbenzene           | 1.0-100.0    | C <sub>9</sub> H <sub>12</sub>                | 103-65-1   |
| Styrene                   | 1.0-100.0    | C <sub>8</sub> H <sub>8</sub>                 | 100-42-5   |
| 1,1,1,2-Tetrachloroethane | 1.0-100.0    | C <sub>2</sub> H <sub>2</sub> Cl <sub>4</sub> | 630-20-6   |
| 1,1,2,2-Tetrachloroethane | 1.0-100.0    | $C_2H_2Cl_4$                                  | 79-34-5    |
| Tetrachloroethene         | 1.0-100.0    | C <sub>2</sub> Cl <sub>4</sub>                | 127-18-4   |
| Toluene                   | 1.0-100.0    | $C_3H_8$                                      | 108-88-3   |
| 1,2,3-Trichlorobenzene    | 1.0-100.0    | $C_6H_3Cl_3$                                  | 87-61-6    |
| 1,2,4-Trichlorobenzene    | 1.0-100.0    | C <sub>6</sub> H <sub>3</sub> Cl <sub>3</sub> | 120-82-1   |
| 1,1,1-Trichloroethane     | 1.0-100.0    | $C_2H_3Cl_3$                                  | 71-55-6    |
| 1,1,2-Trichloroethane     | 1.0-100.0    | $C_2H_3Cl_3$                                  | 79-00-5    |
| Trichloroethene           | 1.0-100.0    | C <sub>2</sub> HCl <sub>3</sub>               | 79-01-6    |
| Trichlorofluoromethane    | 1.0-100.0    | CCl <sub>3</sub> F                            | 75-69-4    |
| 1,2,3-Trichloropropane    | 1.0-100.0    | C <sub>3</sub> H <sub>5</sub> Cl <sub>3</sub> | 96-18-4    |
| 1,2,4-Trimethylbenzene    | 1.0-100.0    | С <sub>9</sub> Н <sub>12</sub>                | 95-63-6    |
| 1,3,5-Trimethylbenzene    | 1.0-100.0    | $C_{9}H_{12}$                                 | 108-67-8   |
| Vinyl chloride            | 1.0-100.0    | C <sub>2</sub> H <sub>3</sub> Cl              | 75-01-4    |
| o-Xylene                  | 1.0-100.0    | $C_8H_8$                                      | 95-47-6    |
| m-Xylene                  | 1.0-100.0    | C <sub>8</sub> H <sub>8</sub>                 | 108-38-3   |
| p-Xylene                  | 1.0-100.0    | $C_8H_8$                                      | 106-42-3   |

# TABLE 2 COMPOUND INFORMATION

### Operational Detection Limit (ODL)

|                             | Waters | Soils          |
|-----------------------------|--------|----------------|
| Compound                    | (ug/l) | <u>(mg/kg)</u> |
| Benzene                     | 1.0    | 0.010          |
| Bromobenzene                | 1.0    | 0.010          |
| Bromochloromethane          | 1.0    | 0.010          |
| Bromodichloromethane        | 1.0    | 0.010          |
| Bromoform                   | 1.0    | 0.010          |
| Bromomethane                | 1.0    | 0.010          |
| n-Butylbenzene              | 1.0    | 0.010          |
| sec-Butylbenzene            | 1.0    | 0.010          |
| tert-Butylbenzene           | 1.0    | 0.010          |
| Carbon tetrachloride        | 1.0    | 0.010          |
| Chlorobenzene               | 1.0    | 0.010          |
| Chloroethane                | 1.0    | 0.010          |
| Chloroform                  | 1.0    | 0.010          |
| Chloromethane               | 1.0    | 0.010          |
| 2-Chlorotoluene             | 1.0    | 0.010          |
| 4-Chlorotoluene             | 1.0    | 0.010          |
| Dibromochloromethane        | 1.0    | 0.010          |
| 1,2-Dibromo-3-chloropropane | 1.0    | 0.010          |
| 1,2-Dibromomethane          | 1.0    | 0.010          |
| Dibromomethane              | 1.0    | 0.010          |
| 1,2-Dichlorobenzene         | 1.0    | 0.010          |
| 1,3-Dichlorobenzene         | 1.0    | 0.010          |
| 1,4-Dichlorobenzene         | 1.0    | 0.010          |
| Dichlorodifluoromethane     | 1.0    | 0.010          |
| 1,1-Dichloroethane          | 1.0    | 0.010          |
| 1,2-Dichloroethane          | 1.0    | 0.010          |
| 1,1-Dichloroethene          | 1.0    | 0.010          |
| cis-1,2-Dichloroethene      | 1.0    | 0.010          |
| trans-1,2-Dichloroethene    | 1.0    | 0.010          |

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# TABLE 2 COMPOUND INFORMATION (Cont.)

# **Operational Detection Limit (ODL)**

| ,                         | Waters        | Soils   |
|---------------------------|---------------|---------|
| Compound                  | <u>(ug/l)</u> | (mg/kg) |
| 2,2-Dichloropropane       | 1.0           | 0.010   |
| 1,1-Dichloropropene       | 1.0           | 0.010   |
| cis-1,3-Dichloropropene   | 1.0           | 0.010   |
| trans-1,3-Dichloropropene | 1.0           | 0.010   |
| Ethylbenzene              | 1.0           | 0.010   |
| Hexachlorobutadiene       | 1.0           | 0.010   |
| Isopropylbenzene          | 1.0           | 0.010   |
| p-Isopropyltoluene        | 1.0           | 0.010   |
| Methylene chloride        | 1.0           | 0.010   |
| Naphthalene               | 1.0           | 0.010   |
| n-Propylbenzene           | 1.0           | 0.010   |
| Styrene                   | 1.0           | 0.010   |
| 1,1,1,2-Tetrachloroethane | 1.0           | 0.010   |
| 1,1,2,2-Tetrachloroethane | 1.0           | 0.010   |
| Tetrachloroethene         | 1.0           | 0.010   |
| Toluene                   | 1.0           | 0.010   |
| 1,2,3-Trichlorobenzene    | 1.0           | 0.010   |
| 1,2,4-Trichlorobenzene    | 1.0           | 0.010   |
| 1,1,1-Trichloroethane     | 1.0           | 0.010   |
| 1,1,2-Trichloroethane     | 1.0           | 0.010   |
| Trichloroethene           | 1.0           | 0.010   |
| Trichlorofluoromethane    | 1.0           | 0.010   |
| 1,2,3-Trichloropropane    | 1.0           | 0.010   |
| 1,2,4-Trimethylbenzene    | 1.0           | 0.010   |
| 1,3,5-Trimethylbenzene    | 1.0           | 0.010   |
| Vinyl chloride            | 1.0           | 0.010   |
| o-Xylene                  | 1.0           | 0.010   |
| m-Xylene                  | 1.0           | 0.010   |
| p-Xylene                  | 1.0           | 0.010   |

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#### **ANALYSIS SUMMARY**

**Gas Chromatograph:** 

• Injector Temp: 175°C

• Detectors: Photoionization Detector (PID) and

Hall Electro Conductivity Detector (HECD) in series

• Detector Temp: PID 250°C

HECD 900°C

HECD Reaction Gas: Hydrogen 25-30 ml/min

• HECD Solvent: n-Propanol 0.5-0.75 ml/min

• Carrier Gas: Helium

• Column: DB-624 75m .53 id capillary

Temperature Program: Held at 35°C for 7 min. then 10°C/min to 90°C, hold for

0.1 min.

Carrier Flow: 10 ml/min Make up flow: 30-50 ml/min

• 1st Alternate Column: Vocol 105m 0.53 id. capillary

Tempeature Program: Hold at 35°C for 4 min, then 4°C/min to 190°C, hold

0.1 min.

Carrier Flow: 10 ml/min Make up flow: 30-50 ml/min

 2nd Alternate Column (for short lists only): 1% SP-1000 on 60/80 Carbopack B, 8' x 0.1" id glass

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Temperature Program: Held at 45°C for 4 min, then 10°C/min to 220°C, hold

for 25 min

Carrier flow: 30 ml/min

#### **Concentrator:**

• Purge Gas: Helium @ 30-40 ml/min

• Purge 11 min

Desorb 4 min @ 180°C

• Bake 4 min at 210°C

**Curve Standards:** 1,2,5,10,100 ppb

Surrogates: 4-Chlorotoluene @ 20 ppb (if not in scan)

MEK @ 20 ppb